INTERNATIONAL STANDARD

ISO 21079-3

First edition
2008-04-15

Chemical analysis of refractories containing alumina, zirconia, and silica — Refractories containing 5 % to 45 % of ZrO_2 (alternative to the X-ray fluorescence method) —

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

Analyse chimique des matériaux réfractaires contenant de l'alumine, de la zircone et de la silice — Matériaux réfractaires contenant de 5 % à 45 % de ZrO_2 (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 21079-3:2008(E)

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Contents

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_2 *(alternative to the X-ray fluorescence method)*:

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

INTERNATIONAL STANDARD **ISO 21079-3:2008(E)**

Chemical analysis of refractories containing alumina, zirconia, and silica — Refractories containing 5 % to 45 % of ZrO_2 (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_2) 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.

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_2 *(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_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.

3.1.2.3 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_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) 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, $w(\mathrm{SiO_2})$, 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.

$$w(\mathrm{SiO_2}) = \frac{(m_1 - m_2) + (m_\mathrm{s} - m_\mathrm{b}) \times \frac{500}{10}}{m} \times 100 \qquad (1)$$

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_2	0,1 to 10
Al_2O_3	1 to 10
Fe_2O_3	0,01 to 2
TiO_2	0,01 to 5
CaO	0,01 to 2
MgO	0,01 to 2
Cr_2O_3	0,01 to 3
HfO_2	0,01 to 2

NOTE 1 When solutions (S1) or (S'1) are 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 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_2 0,1 mg/ml, Al_2O_3 0,10 mg/ml, Fe_2O_3 0,02 mg/ml, CaO 0,02 mg/ml, MgO 0,2 mg/ml, HfO_2 0,02 mg/ml, Cr_2O_3 0,03 mg/ml and TiO_2 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	Internal standard solution	Mixed standard solution 2	Concentration of solution mg/100 ml							
	ml	ml	ml	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	Cr_2O_3	HfO_2
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.

Table 4 — Examples 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
CaO	Ca	393,366
MgO	Mg	279,553
Cr_2O_3	Cr	267,716
HfO_2	Hf	277,336
Internal standard element	Y	371,030
	Sc	361,383

3.2.4 Blank test

Transfer a 10 ml aliquot portion of blank solution (B1), (B’1) or (B’’1) (see Annex A of ISO 21079-1:2008) and carry out the procedure given in 3.2.3. Designate the solution corresponding to stock solution (B1), (B’1) or (B’’1) as blank solution (B1dScY), (B’1dScY) 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 relation between the emission intensity and mass of each component (SiO_2, Al_2O_3, Fe_2O_3, TiO_2 , CaO, MgO, Cr_2O_3 and HfO_2). Prepare the calibration graph for each component.

3.2.6 Calculation

Calculate the mass fraction of each component $w(SiO_2)$, $w(Al_2O_3)$, $w(Fe_2O_3)$, $w(TiO_2)$, $w(CaO)$, $w(MgO)$, $w(Cr_2O_3)$ and $w(HfO_2)$, indicated as $w(M_nO_m)$, as a percentage, using Equation (2), with iron(III) oxide that is derived from the emission intensity in 3.2.3 and 3.2.4, and the calibration in 3.2.5.

$$w\left(\mathrm{M}_n\mathrm{O}_m\right) = \frac{m_\mathrm{s} - m_\mathrm{b}}{m} \times \frac{500}{20} \times 100 \tag{2}$$

where

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

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

m is the mass of the test portion, in accordance with 9.3.2.2, 9.3.3.2 or 9.3.4.2 of ISO 21079-1:2008, in grams.

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

3.3.1 Principle

The emission intensity of calcium, magnesium, sodium and potassium in stock solution (S4) is measured by the ICP-AE spectrometer.

3.3.2 Reagents

3.3.2.1 General

Prepare the reagents given in 3.3.2.2 and 3.3.2.3 and those specified in ISO 26845, as necessary.

3.3.2.2 Mixed standard solution 3, CaO 0,10 mg/ml, MgO 0,10 mg/ml, Na_2O 0,10 mg/ml, and K_2O 0,10 mg/ml.

Transfer 50 ml each of the calcium oxide, the magnesium, the sodium oxide and the potassium oxide standard solutions into 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 3 to each of several 100 ml volumetric flasks. To each, add 5 ml of hydrochloric acid (1+1), and dilute to the mark with water.

Typical examples of preparation are shown in Table 5.

Table 5 — Example of series 4 solution for calibration

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

3.3.3 Procedure

Spray a portion of stock solution (S4) (see Annex A of ISO 21079-1:2008) into the argon plasma flame of an ICP-AE spectrometer, and measure the emission intensity of each element at the appropriate wavelength.

NOTE Examples of appropriate wavelengths are given in Table 6.

Table 6 — Example of wavelength

Component	Element	Wavelength nm
CaO	Ca	393,366
MgO	Mg	279,553
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 Annex A of ISO 21079-1:2008).

3.3.5 Plotting the calibration graph

Carry out the procedure described in 3.3.3 using Sseries 4 solutions for calibration. Plot the relation between the emission intensity and mass of each component (CaO, MgO, Na_2O and K_2O). Prepare the calibration graph for each component.

3.3.6 Calculation

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

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

where

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

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

m is the mass of the test portion prepared in accordance with 9.3.4.2 of ISO 21079-1:2008, in grams.

4 Instrumental methods using Flame Atomic Absorption Spectrometry (FAAS)

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

4.1.1 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 absorbance of calcium, magnesium, sodium and potassium is measured.

4.1.2 Reagents

4.1.2.1 General

Prepare the reagents given in 4.1.2.2 and 4.1.2.3 and those specified in ISO 26845, as necessary.

4.1.2.2 Mixed standard solution 3, CaO 0,10 mg/ml, MgO 0,10 mg/ml, Na_2O 0,10 mg/ml, and K_2O 0,10 mg/ml.

Prepare as described in 3.3.2.2.

4.1.2.3 Series 5 solution for calibration.

Transfer aliquot portions of mixed standard solution 3 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.

Typical examples of solutions for calibration are shown in Table 7.

Table 7 — Example of series 5 solution for calibration

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

4.1.3 Procedure

Spray a portion of stock solution (S3) (see Annex A ISO 21079-1:2008) into the flame of an atomic absorption (AA) spectrometer, and measure the absorption of each element at the appropriate wavelength.

Examples of wavelengths are given in Table 8.

Table 8 — Examples of wavelength

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

NOTE There can be samples where the mass fractions of the oxides found are greater than those given in the scope of ISO 21079-1:2008. In the absence of another suitable standard procedure, it will be necessary to transfer an appropriate volume (x ml) of stock solution (S3) into a 100 ml volumetric flask. Then add $5{,}0-5{,}0\times\left(\frac{x}{100}\right)$ ml of hydrochloric acid (1+1) and $10{,}0-10{,}0\times\left(\frac{x}{100}\right)$ ml of lanthanum solution. Dilute to the mark with water, and measure with this solution.

4.1.4 Blank test

Carry out the procedure described in 4.1.3 using blank solution (B3) (see Annex A of ISO 21079-1:2008).

NOTE There can be samples where the mass fractions of the oxides found are greater than those given in the scope of ISO 21079-1:2008. In the absence of another suitable standard procedure, the blank solution (B3) is prepared using the same procedure as that for the stock solution in the note to 4.1.3.

4.1.5 Plotting the calibration graph

Carry out the procedure described in 4.1.3 using series 5 solution for calibration, as described in 4.1.2.3. Plot the relation between the absorbance and mass of each component (CaO, MgO, Na_2O and K_2O). Prepare the calibration graph for each component.

4.1.6 Calculation

Calculate the mass fraction of each component $w(CaO)$, $w(MgO)$, $w(Na_2O)$ and $w(K_2O)$, indicated as $w(M_nO_m)$, expressed as a percentage, using Equation (4). Use the mass of calcium oxide derived from the absorbance obtained as described in 4.1.3 and 4.1.4, and the calibration prepared as described in 4.1.5.

$$w\left(M_nO_m\right)=\frac{m_s-m_b}{m}\times\frac{100}{V}\times 100 \tag{4}$$

where

m_s is the mass of each component, indicated as M_nO_m, in the aliquot portion of stock solution (S3) or the diluted stock solution, in grams;

m_b is the mass of each component, indicated as M_nO_m, in the aliquot portion of blank solution (B3) or the diluted blank solution, in grams;

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

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

m is the mass of the test portion obtained in accordance with 9.3.3.2 of ISO 21079-1:2008, in grams.

4.2 Determination of chromium(III) oxide by FAAS spectrometry

4.2.1 Principle

The absorbance of chromium(III) oxide is measured in an aliquot portion of stock solution (S1) or (S'1) by an atomic absorption spectrometer.

4.2.2 Reagents

4.2.2.1 General

Prepare the reagents given in 4.2.2.2 to 4.2.2.5 and those specified in ISO 26845, as necessary.

4.2.2.2 Any reagents specified in ISO 21079-1, as necessary.

4.2.2.3 Diluted chromium oxide standard solution, Cr_2O_3 0,02 mg/ml.

Measure 10 ml of chromium(III) oxide (1 mg/ml) standard solution into a 500 ml volumetric flask, and dilute to the mark with water.

4.2.2.4 Matrix solution 1, 1' or 1'', prepared as described in 3.2.2.6.

4.2.2.5 Series 6 solutions for calibration.

Transfer aliquot portions of diluted chromium oxide standard solution to several 100 ml volumetric flasks. To each, add 10 ml of lanthanum solution and 20 ml of matrix solution 1, 1' or 1'', 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 solutions for calibration

Solution No.	Matrix solution 1, 1' or 1'' ml	Lanthanum solution ml	Diluted chromium oxide standard solution ml	Concentration of solution Cr_2O_3 mg/100 ml
1	20	10	0	0,00
2	20	10	1	0,02
3	20	10	2	0,04
4	20	10	3	0,06
5	20	10	4	0,08
6	20	10	5	0,10
7	20	10	10	0,20
8	20	10	15	0,30
9	20	10	20	0,40

NOTE Matrix solution 1 is used for stock solution (S1), matrix solution 1' is used for stock solution (S'1) and matrix solution 1'' is used for stock solution (S''1).

4.2.3 Procedure

Transfer a 20 ml aliquot portion of stock solution (S1), (S'1) or (S''1) (see Annex A of ISO 21079-1:2008) to a 100 ml volumetric flask, add 10 ml of lanthanum solution, and dilute to the mark with water. Designate this solution as stock solution (S1dLa), stock solution (S'1dLa) or stock solution (S''1dLa).

Spray a portion of stock solution (S1dLa), (S'1dLa) or (S''1dLa) into the dinitrogen oxide/acetylene flame of an AA spectrometer, measure the absorbance at a wavelength of 357,9 nm.

4.2.4 Blank test

Carry out the procedure described in 4.2.3 with blank solution (B1), (B'1) or (B''1) (see Annex A of ISO 21079-1:2008). Designate the solution corresponding to stock solutions (S1dLa), (S'1dLa) or (S''1dLa) as blank solution (B1dLa), (B'1dLa) or (B''1dLa).

4.2.5 Plotting the calibration graph

Carry out the procedure described in 4.2.3 using series 6 solutions for calibration as given in 4.2.2.5. Plot the relation between the absorbance and mass of chromium(III) oxide.

4.2.6 Calculation

Calculate the mass fraction of chromium(III) oxide, $w(\mathrm{Cr_2O_3})$, expressed as a percentage, using Equation (5) with the mass of chromium(III) oxide which is derived from the absorbance described in 4.2.3 and 4.2.4, and the calibration prepared as described in 4.2.5.

$$w\left(\mathrm{Cr_2O_3}\right) = \frac{m_\mathrm{s} - m_\mathrm{b}}{m} \times \frac{500}{20} \times 100 \qquad (5)$$

where

m_s is the mass of chromium(III) oxide in the aliquot portion of stock solution (S1dLa), (S’1dLa) or (S’’1dLa), in grams;

m_b is the mass of chromium(III) oxide in the aliquot portion of blank solution (B1dLa), (B’1dLa) or (B’’1dLa), in grams;

m is the mass of the test portion given in 9.2.2.2, 9.2.3.2 or 9.2.4.2 of ISO 21079-1:2008, in grams.

5 Calculation and expression of test results

Express the test results in accordance with ISO 26845.

6 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 71.040.40; 81.080

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