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Chemical analysis of magnesite and dolomite refractory products (alternative to the X-ray fluorescence method) —

Part 3:

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

Analyse chimique des produits de magnésie et de dolomie (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 10058-3:2008(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 10058-3 was prepared by Technical Committee ISO/TC 33, *Refractories*.

This first edition of ISO 10058-3, together with ISO 10058-1 and ISO 10058-2, cancels and replaces ISO 10058:1992 which has been technically revised to include the increasing use of flame atomic absorption spectrophotometry (FAAS) and inductively coupled plasma atomic emission spectrometry (ICP-AES) methods as well some improvements in the wet chemical analyses procedures developed in Japan.

ISO 10058 consists of the following parts, under the general title *Chemical analysis of magnesite and dolomite refractory products (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 spectrophotometry (FAAS) and inductively coupled plasma atomic emission spectrometry (ICP-AES)*

Chemical analysis of magnesite and dolomite refractory products (alternative to the X-ray fluorescence method) —

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

1 Scope

This part of ISO 10058 specifies atomic absorption spectrometry (AAS) and inductively coupled plasma atomic emission spectrometry (ICP-AES) methods for the chemical analysis of magnesite and dolomite refractory products and raw materials.

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

Table 1 — Range of determination (percentage by mass)

Component	Range	Component	Range
SiO_2	0,1 to 10	Na_2O	0,01 to 1
Al_2O_3	0,05 to 10	K_2O	0,01 to 1
Fe_2O_3	0,01 to 10	Cr_2O_3	0,01 to 3
TiO_2	0,01 to 1	ZrO_2	0,01 to 1
MnO	0,01 to 1	P_2O_5	0,01 to 5
CaO	0,01 to 10	—	—
LOI	0,01 to 60	—	—
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 10058-1:2008, *Chemical analysis of magnesite and dolomite refractory products (alternative to the X-ray fluorescence method) — Part 1: Apparatus, reagents, dissolution and 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 ICP-AES

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

3.1.1 Principle

The residual silica remaining in solutions (S1) (see ISO 10058-1:2008, Annex A) is determined using ICP-AES.

3.1.2 Reagents

3.1.2.1 Series 1 solution for calibration.

Transfer appropriate amounts of aliquot portions of dilute silicon(IV) oxide standard solution (0,08 mg/ml) precisely to several 100 ml volumetric flasks in accordance with the composition of the samples. To each, add 10 ml of matrix solution 1 and dilute to the mark with water. An example is shown in Table 2.

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

Table 2 — Example of series 1 solution for calibration

Series 1 solution No.	Matrix solution 1 ml	Dilute 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) as follows.

Transfer 10 ml of stock solution (S1) to a 100 ml volumetric flask and dilute to the mark with water.

This solution, which is used for the determination of dissolved silicon(IV) oxide, is designated 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 a wavelength of 251,611 nm.

3.1.4 Blank test

Carry out the procedure in 3.1.3 with blank solution (B1) (see ISO 10058-1:2008, Annex A).

The equivalent diluted blank solution to diluted stock solution (S1d) is designated as diluted blank solution (B1d).

3.1.5 Plotting the calibration graph

Using the series 1 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.

Carry out a new calibration 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), from the amount 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:

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

where

m is the mass of the test portion calculated in accordance with ISO 10058-1:2008, 8.2.2.3.1, in grams (g);

m_1 is the mass from ISO 10058-1:2008, 8.2.2.3.3, in grams (g);

m_2 is the mass from ISO 10058-1:2008, 8.2.2.4, 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, chromium(III) oxide and zirconium oxide using stock solutions (S1) 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, manganese(II) oxide, calcium oxide, chromium(III) oxide and zirconium oxide is measured by ICP-AE spectrometer for stock solutions (S1) or (S′1) (see ISO 10058-1:2008, Annex A). This method should be applied to components in (S1) or (S′1) in accordance with Table 3.

Table 3 — Range of determination (percentage by mass)

Component	Range	Component	Range
SiO_2	0,1 to 10	MnO	0,01 to 1
Al_2O_3	0,05 to 10	CaO	0,01 to 10
Fe_2O_3	0,01 to 10	Cr_2O_3	0,01 to 3
TiO_2	0,01 to 1	ZrO_2	0,01 to 1

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 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

Reagents given in ISO 10058-1:2008, Clause 5, and the following.

3.2.2.1 Mixed standard solution 1, 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 0,02 mg/ml, ZrO_2 0,005 mg/ml.

Transfer an aliquot portion (1 mg/ml) 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 (20 ml) and zirconium oxide (5 ml) solutions into a 1 000 ml volumetric flask and dilute to the mark with water.

3.2.2.2 Matrix solution 2 or 2′.

Carry out the procedure given in ISO 10058-1:2008, 8.2.2.3 or 8.2.3.3 without the sample, but omit heating the fusion mixture or anhydrous sodium carbonate.

The equivalent solution to stock solution (S1) or (S′1) is designated as matrix solution 2 or 2′ as applicable.

3.2.2.3 Internal standard solution.

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

3.2.2.4 Series 2 solutions for calibration.

Transfer aliquot portions, determined using Table 4, of mixed standard solution 1 to each of several 100 ml volumetric flasks. Add 10 ml of matrix solution 2 or 2′ and 5 ml of internal standard solution, respectively, and dilute to the mark with water.

Table 4 gives examples of the preparation of solutions. 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 4 — Example of series 2 solution for calibration

Series 2 solution	Matrix solution 2 or 2′	Internal standard solution	Mixed standard solution 1	Concentration of solution mg/100 ml							
No.	ml	ml	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	0,04	0,010
3	10	5	5	0,20	0,20	0,20	0,025	0,025	0,20	0,10	0,025
4	10	5	10	0,40	0,40	0,40	0,050	0,050	0,40	0,20	0,050
5	10	5	20	0,80	0,80	0,80	0,100	0,100	0,80	0,40	0,100
6	10	5	30	1,20	1,20	1,20	0,150	0,150	1,20	0,60	0,150
7	10	5	40	1,60	1,60	1,60	0,200	0,200	1,60	0,80	0,200
8	10	5	50	2,00	2,00	2,00	0,250	0,250	2,00	1,00	0,250

NOTE 1 In this table, an example of the preparation of solutions is shown. In accordance with the compositions of the samples and the types and capabilities of the instrument used, an appropriate solution series for calibration is prepared.

NOTE 2 To use this approach to calibration, it is essential that line interferences of any of these oxides on each other be checked for and, if present, that appropriate corrections be applied.

3.2.3 Procedure

Transfer a 10 ml aliquot portion of stock solution (S1) or (S′1) into 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 a wavelength determined in accordance with 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

Transfer a 10 ml aliquot portion of blank solution (B1) or (B′1) (see ISO 10058-1:2008, Annex A) and carry out the procedure given in 3.2.3. The solution corresponding to stock solution (B1) or (B′1) is designated as blank solution (B1dScY) or (B′1dScY).

3.2.5 Plotting the calibration graph

Use series 2 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, MnO, CaO, Cr_2O_3 and ZrO_2). Prepare the calibration graph for each component.

3.2.6 Calculation

Calculate the mass fraction 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 $w_{\mathrm{ZrO_2}}$), indicated as $w_{\mathrm{M}_n\mathrm{O}_m}$ and expressed as a percentage, using Equation (2), and derived from the emission intensity in 3.2.3 and 3.2.4, and the calibration in 3.2.5.

$$w_{\mathrm{M}_n\mathrm{O}_m} = \frac{m_\mathrm{s} - m_\mathrm{b}}{m} \times \frac{250}{10} \times 100 \qquad (2)$$

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 in ISO 10058-1:2008, 8.2.2.3.1 or 8.2.3.3, 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 10058-1:2008, Annex A) is measured by an ICP-AE spectrometer.

3.3.2 Reagents

Use reagents given in ISO 10058-1 and the following.

3.3.2.1 Matrix solution 3.

Transfer adequate 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 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.

3.3.2.2 Mixed standard solution 2, Na_2O 0,05 mg/ml, K_2O 0,05 mg/ml.

Transfer 25 ml of the standard sodium oxide and potassium oxide solutions (both 1 mg/ml) to a 500 ml volumetric flask and dilute to the mark with water.

3.3.2.3 Series 3 solution for calibration.

Transfer aliquot portions of mixed standard solution 2 to each of several 100 ml volumetric flasks. To each, add 20 ml of matrix solution 3 and 5 ml of hydrochloric acid (1+1) and dilute to the mark with water.

Typical examples of preparation are shown in Table 6.

Table 6 — Example of series 3 solution for calibration

Series 3 solution	**Matrix solution 3**	**Hydrochloric acid (1+1)**	**Mixed standard solution 2**	**Concentration of solution** mg/100 ml	
No.	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) into the argon plasma flame of an ICP-AE spectrometer, and measure the emission intensity of each of the elements at the wavelength given in Table 7.

Table 7 — 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 10058-1:2008, Annex A).

3.3.5 Plotting the calibration graph

Carry out the procedure described in 3.3.3 using series 3 solutions for calibration. Plot the relation between the emission intensity and mass of each component (Na_2O and K_2O). Prepare the calibration graph for each component.

3.3.6 Calculation

Calculate the mass fraction of each component (sodium oxide, $w_{\mathrm{Na_2O}}$, and potassium oxide, $w_{\mathrm{K_2O}}$), indicated as $w_{\mathrm{M}_n\mathrm{O}_m}$ and expressed as a percentage, using Equation (3). Use the mass of the respective 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 the component, indicated as $\mathrm{M}_n\mathrm{O}_m$, in stock solution (S4), in grams (g);

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

m is the mass of the test portion prepared in ISO 10058-1:2008, 8.3.4.3, 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 an ICP-AE spectrometer on stock solution (S1dScY or S′1dScY) (3.2.3).

3.4.2 Reagents

Reagents given in ISO 10058-1:2008, Clause 5, and the following.

3.4.2.1 Matrix solution 4.

Transfer adequate 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.

Prepare as described in the note in 3.3.2.1.

3.4.2.2 **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. 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) into a 100 ml volumetric flask and dilute to the mark with water.

3.4.2.4 Series 4 solution for calibration. Transfer aliquot portions of dilute phosphorus(V) oxide standard solution (P_2O_5 0,04 mg/ml) to each of several 100 ml volumetric flasks. To each, add 10 ml of matrix solution 4, the appropriate amount of aluminium oxide solution, 5 ml of internal standard solution (3.4.2.3) and 5 ml of hydrochloric acid (1+1), and dilute to the mark with water.

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

In the series 2 solution for calibration, standard phosphorus(V) oxide solution may be added instead of the standard zirconium oxide solution.

Table 8 — Example of the series 4 solution for calibration

Series 4 solution No.	Matrix solution 4 ml	Aluminium oxide solution [a] (1 mg/ml) ml	Internal standard solution ml	Dilute standard phosphorus(V) oxide solution ml	Concentration of solution P_2O_5 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 content percentage of aluminium oxide is 5 % by mass.

3.4.3 Procedure

Spray a portion of the solution (S1dScY or S1′dScY) obtained in 3.2.3 into the argon plasma flame of an ICP-AE spectrometer, and measure the emission intensity at a wavelength of 213,620 nm, for example. 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 4 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, $w_{P_2O_5}$, expressed as a percentage, using Equation (4), with 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.

$$w_{P_2O_5} = \frac{m_s - m_b}{m} \times \frac{250}{V} \times 100 \quad (4)$$

where

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

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

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

m is the mass of the test portion in ISO 10058-1:2008, 8.2.2.3.1 or 8.2.3.3, in grams (g).

4 Instrumental methods using FAAS

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

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 10058-1:2008, Annex A) by an AA spectrometer.

4.1.2 Reagents

Use reagents given in ISO 10058-1:2008, Clause 5, and the following.

4.1.2.1 Matrix solution 2 or 2′, prepared as described in 3.2.2.2.

4.1.2.2 Mixed standard solution 3, MnO 0,02 mg/ml, CaO 0,10 mg/ml, Cr_2O_3 0,04 mg/ml. Transfer an aliquot of standard (1 mg/ml) manganese(II) oxide (10 ml), calcium oxide (50 ml), and chromium(III) oxide (20 ml) solutions into a 500 ml volumetric flask and dilute to the mark with water.

4.1.2.3 Standard lanthanum solution, La 1,0 mg/ml. Dry about 2 g of lanthanum oxide at 110 °C ± 5 °C for 60 min, cool in a desiccator. Weigh 1,172 8 g of this, transfer to a 600 ml beaker. Dissolve by gradually adding 100 ml of hydrochloric acid (1+1) and dilute precisely to 1 000 ml in a volumetric flask with water.

4.1.2.4 Series 5 solutions for calibration. Transfer aliquot portions of mixed standard solution 3 (4.1.2.2) to several 100 ml volumetric flasks. To each, add 10 ml of lanthanum solution (4.1.2.3) and 20 ml of matrix solution 5 or 5′, and dilute to the mark with water.

A typical example of solutions for calibration is shown in Table 9.

Table 9 — Example of series 5 solution for calibration

Series 5 solution No.	Matrix solution 5 or 5′ ml	Lanthanum solution ml	Mixed standard solution 3 ml	Concentration of solution mg/100 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 2 is used for stock solution (S1); matrix solution 2′ is used for stock solution (S′1).

4.1.3 Procedure

Transfer a 20 ml aliquot portion of stock solution (S1) or (S′1) to a 100 ml volumetric flask, add 10 ml of lanthanum solution (4.1.2.3), 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 FAA spectrophotometer, measure the absorbance of each element at the appropriate wavelength 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 10058-1:2008, Annex A). The solution corresponding to stock solutions (S1dLa) or (S′1dLa) is designated as blank solution (B1dLa) or (B′1dLa).

4.1.5 Plotting the calibration graph

Carry out the procedure described in 4.1.3 using series 5 calibration solutions for calibration. Plot the relation between the absorbance and mass of each component (MnO, CaO and Cr_2O_3). Prepare the calibration graph for each component.

4.1.6 Calculation

Calculate the mass fraction of each component (manganese oxide, w_{MnO}, calcium oxide, w_{CaO}, and chromium(III) oxide, $w_{\mathrm{Cr_2O_3}}$), indicated as $w_{\mathrm{M}_n\mathrm{O}_m}$ and expressed as a percentage, using Equation (5), with the mass of the respective oxide which is derived from the absorbance described in item 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), 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), in grams (g);

m is the mass of the test portion described in ISO 10058-1:2008, 8.2.2.3.1 or 8.2.3.3, 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 FAA spectrophotometer, and the emission intensity of calcium, sodium and potassium is measured.

4.2.2 Reagents

Use reagents given in ISO 10058-1 and the following.

4.2.2.1 Mixed standard solution 4, 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 (1 mg/ml), the sodium oxide (1 mg/ml) and the potassium oxide (1 mg/ml) into a 500 ml volumetric flask and dilute to the mark with water.

4.2.2.2 Series 6 solution for calibration.

Transfer aliquot portions of mixed standard solution 4 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 11.

Table 11 — Example of series 6 solution for calibration

Series 6 solution No.	Hydrochloric acid (1+1) ml	Lanthanum solution ml	Mixed standard solution 4 ml	Concentration of solution mg/100 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) prepared in ISO 10058-1:2008, 8.3.3.3, into the dinitrogen oxide-acetylene flame of an FAA spectrophotometer, measure the absorption of each element at a wavelength determined in accordance with Table 12.

Table 12 — Example of wavelength

Component	Element	Wavelength nm
CaO	Ca	422,7
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 (4.1.2.3). 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) prepared in ISO 10058-1:2008, 8.3.3.4.

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 the calibration graph

Carry out the procedure described in 4.2.3 using series 6 calibration solutions for calibration. Plot the relation between the absorbance and mass of each component (CaO, Na_2O and K_2O). Prepare the calibration graph for each component.

4.2.6 Calculation

Calculate the mass fraction of each component (calcium oxide, w_{CaO}, sodium oxide, $w_{\mathrm{Na_2O}}$, and potassium oxide, $w_{\mathrm{K_2O}}$), indicated as $w_{\mathrm{M}_n\mathrm{O}_m}$ and expressed as a percentage, using Equation (6). Use the mass of the respective oxide 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 prepared in ISO 10058-1:2008, 8.3.3.3, in grams (g).

5 Test report

Prepare a test report as described in ISO 26845.

ICS 73.080

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