
**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 magnésite et de dolomite (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)*



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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

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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 ₂ O	0,01 to 1
Al ₂ O ₃	0,05 to 10	K ₂ O	0,01 to 1
Fe ₂ O ₃	0,01 to 10	Cr ₂ O ₃	0,01 to 3
TiO ₂	0,01 to 1	ZrO ₂	0,01 to 1
MnO	0,01 to 1	P ₂ O 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_{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:

$$w_{\text{SiO}_2} = \frac{(m_1 - m_2) + (m_s - m_b) \times \frac{250}{10}}{m} \times 100 \quad (1)$$

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 ₂ O ₃	0,05 to 10	CaO	0,01 to 10
Fe ₂ O ₃	0,01 to 10	Cr ₂ O 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 SiO₂ is residual silica. When solution (S'1) is used, the SiO₂ 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, SiO₂ 0,04 mg/ml, Al₂O₃ 0,04 mg/ml, Fe₂O₃ 0,04 mg/ml, TiO₂ 0,005 mg/ml, MnO 0,005 mg/ml, CaO 0,04 mg/ml, Cr₂O₃ 0,02 mg/ml, ZrO₂ 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 No.	Matrix solution 2 or 2' ml	Internal standard solution ml	Mixed standard solution 1 ml	Concentration of solution mg/100 ml							
				SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	MnO	CaO	Cr ₂ O 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 (S'1'dScY).

Spray a portion of stock solution (S1dScY) or (S'1'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 ₂ O ₃	Al	396,152
Fe ₂ O ₃	Fe	259,940
TiO ₂	Ti	334,941
MnO	Mn	257,610
CaO	Ca	393,366
Cr ₂ O ₃	Cr	267,716
ZrO ₂	Zr	343,823
Internal standard element	Y	371,030
	Sc	361,383

3.2.4 Blank test

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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'1'dScY).

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 (SiO₂, Al₂O₃, Fe₂O₃, TiO₂, MnO, CaO, Cr₂O₃ and ZrO₂). Prepare the calibration graph for each component.

3.2.6 Calculation

Calculate the mass fraction of each component (w_{SiO_2} , $w_{\text{Al}_2\text{O}_3}$, $w_{\text{Fe}_2\text{O}_3}$, w_{TiO_2} , w_{MnO} , w_{CaO} , $w_{\text{Cr}_2\text{O}_3}$ and w_{ZrO_2}), indicated as $w_{M_nO_m}$ 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.

$$w_{M_nO_m} = \frac{m_s - m_b}{m} \times \frac{250}{10} \times 100 \quad (2)$$

where

m_s is the mass of each component, indicated as M_nO_m , in stock solution (S1dScY) or (S'1'dScY), in grams (g);

m_b is the mass of each component, indicated as M_nO_m , in blank solution (B1dScY) or (B'1'dScY), 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).