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

**Part 2:
Wet chemical analysis**

*Analyse chimique des produits de magnésite et de dolomie (méthode
alternative à la méthode par fluorescence de rayons X) —
Partie 2: Méthodes d'analyse chimique par voie humide*

ISO 10058-2:2008

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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 10058-2 was prepared by Technical Committee ISO/TC 33, *Refractories*.

This first edition of ISO 10058-2, together with ISO 10058-1 and ISO 10058-3, 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 2: Wet chemical analysis

1 Scope

This part of ISO 10058 specifies traditional (“wet process”) 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	MgO	30 to 99,9
Al ₂ O ₃	0,05 to 10	Na ₂ O	0,01 to 1
Fe ₂ O ₃	0,01 to 10	K ₂ O	0,01 to 1
TiO ₂	0,01 to 1	Cr ₂ O ₃	0,01 to 3
MnO	0,01 to 1	ZrO ₂	0,01 to 1
CaO	0,01 to 60	P ₂ O ₅	0,01 to 5
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 determination of gravimetric silica*

ISO 10058-3:2008, *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 atomic plasma emission spectrometry (ICP-AES)*

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 Determination of silicon(IV) oxide

3.1 General

The determination of silicon(IV) oxide is carried out using one of the following methods.

- a) Combined use of the dehydration or the coagulation and molybdenum blue methods

This method is applied to samples consisting of more than 4 % by mass of silicon(IV) oxide (see 3.2).

- b) Molybdenum blue method

This method is applied to samples consisting of less than 10 % by mass of silicon(IV) oxide (see 3.3).

3.2 Combined use of the coagulation and molybdenum blue methods

3.2.1 Principle

An aliquot portion of the stock solution (S1) (see ISO 10058-1:2008, Annex A), after pH adjustment, is treated with ammonium molybdate and the silicomolybdate is reduced to yield molybdenum blue, the absorbance of which is measured.

The sum of this residual silicon(IV) oxide in solution plus the mass of silicon (IV) oxide ($m_1 - m_2$) calculated in accordance with ISO 10058-1:2008, 8.2.2.3.3, gives the total silicon(IV) oxide content.

3.2.2 Procedure

This determination should be commenced with little delay after the stock solution (S1) is prepared, as prolonged standing may allow polymerization of silica to occur leading to low results.

Transfer 10 ml of stock solution (S1) (see ISO 10058-1:2008, Annex A) to a 100 ml plastic beaker, add 2 ml of hydrofluoric acid (1+9) and mix with a plastic rod. Allow to stand for 10 min and add 50 ml of boric acid solution. Add 2 ml of ammonium molybdate solution while mixing at a temperature of 25 °C and allow to stand for 10 min. Add 5 ml of L (+)-tartaric acid solution while stirring and, after 1 min, add 2 ml of L (+)-ascorbic acid solution. Transfer the solution to a 100 ml volumetric flask, dilute to the mark with water, mix and allow to stand for 60 min.

Measure the absorbance of the solution in a 10 mm cell at a wavelength of 650 nm using water as reference.

3.2.3 Plotting the calibration graph

Transfer 0 ml, 2 ml, 4 ml, 6 ml, 8 ml and 10 ml aliquot portions [0 mg to 0,4 mg as silicon(IV) oxide] of dilute standard silicon(IV) oxide solution (SiO_2 0,04 mg/ml) to separate 100 ml plastic beakers and add to each 10 ml of blank solution (B1) (see ISO 10058-1:2008, Annex A). Treat these solutions and measure the absorbance as given in 3.2.2, and plot the absorbances against the amounts of silicon(IV) oxide. Prepare the calibration graph by adjusting the curve so that it passes through the point of origin.

3.2.4 Blank test

Using the blank solution (B1) (see ISO 10058-1:2008, Annex A), carry out the procedure given in 3.2.2.

3.2.5 Calculation

Calculate the mass fraction of silicon(IV) oxide, w_{SiO_2} , expressed as a percentage, using Equation (1), with the absorbances obtained by the procedures given in 3.2.2 and 3.2.4 and the calibration graph plotted in 3.2.3.

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

where

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 10058-12008, 8.2.2.3.3, in grams (g);

m_s is the mass of silicon(IV) oxide in the aliquot portion of stock solution (S1) as applicable, in grams (g);

m_b is the mass of silicon(IV) oxide in the aliquot portion of blank solution (B1) as applicable, in grams (g);

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

3.3 Molybdenum blue method

3.3.1 Principle

An aliquot portion of the stock solution (S'1) (see ISO 10058-1:2008, Annex A), after pH adjustment, is treated with ammonium molybdate and the silicomolybdate is reduced to yield molybdenum blue, the absorbance of which is measured.

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3.3.2 Procedure <https://standards.iteh.ai/catalog/standards/sist/ddcfac00-199c-4580-8171-1f8303b3ae95/iso-10058-2-2008>

Transfer precisely an aliquot portion of stock solution (S'1) (see ISO 10058-1:2008, Annex A) to two 100 ml plastic beakers and add to each an aliquot portion of blank solution (B'1) (see ISO 10058-1:2008, Annex A). Add to each 2 ml of hydrofluoric acid (1+9), mix with a plastics rod and allow to stand for 10 min. Add 50 ml of boric acid solution, dilute to 80 ml with water. Add 5 ml of ammonium molybdate solution while mixing at a temperature of 25 °C and allow to stand for 10 min. Add 5 ml of L (+)-tartaric acid solution while stirring and, after 1 min, add to 10 ml of L (+)-ascorbic acid solution. Transfer each solution to a 200 ml volumetric flask, dilute to the mark with water and mix. Allow to stand for 60 min and measure the absorbance of the solutions in a 10 mm cell at a wavelength of 650 nm against water as a reference. Average the two measurements.

NOTE Aliquot volumes of stock solution (S'1) and blank solution (B'1) corresponding to the mass fraction of silicon(IV) oxide in the sample are shown in Table 2.

If the difference of the two absorbance measurements is greater than 0,005, repeat the procedure given in 3.3.2 and ISO 10058-1:2008, 8.2.3.3. When measurements of the same sample with around 1,0 absorbance are repeated, it is necessary for the spectrophotometer to show the differences within 0,002.

Table 2 — Aliquot volumes of stock solution (S'1) and blank solution (B'1)

Mass fraction of silicon(IV) oxide %	Aliquot volume of stock solution (S'1) ml	Aliquot volume of blank solution (B'1) ml
< 2	20	0
≥ 2, < 4	10	10
≥ 4, ≤ 10	5	15

3.3.3 Blank test

Carry out a blank determination by treating the blank solution (B1) (see ISO 10058-1:2008, Annex A) and following the procedure described into 3.3.2. The volume of the aliquot portion of blank solution is the same as those for the corresponding "S" solution.

3.3.4 Plotting of calibration graph

Transfer 0 ml, 5 ml, 10 ml, 15 ml, 20 ml and 25 ml aliquot portions [0 mg to 1 mg as silicon(IV) oxide] of dilute standard silicon(IV) oxide solution (SiO₂ 0,04 mg/ml) to separate 100 ml plastic beakers and add to each 20 ml of blank solution (B'1) (see ISO 10058-1:2008, Annex A). Treat these solutions and measure the absorbance in accordance with the procedure from the addition of hydrofluoric acid (1+9) in 3.3.2. Plot the absorbance against the amounts of silicon(IV) oxide and prepare the calibration graph by adjusting the curve so that it passes through the point of origin.

3.3.5 Calculation

Calculate the mass fraction of silicon(IV) oxide, w_{SiO_2} , expressed as a percentage, using Equation (2), with the amount of silicon(IV) oxide derived from the absorbance measurements obtained in 3.3.2 and 3.3.3 and the calibration in 3.3.4.

$$w_{SiO_2} = \frac{m_s - m_b}{m} \times \frac{250}{V} \times 100 \tag{2}$$

where

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m_s is the mass of silicon(IV) oxide in the aliquot portion of stock solution (S'1), in grams (g);

m_b is the mass of silicon(IV) oxide in the aliquot portion of blank solution (B'1), in grams (g);

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V is the aliquot portion volume of stock solution (S'1), in millilitres (ml);

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

4 Determination of aluminium oxide

4.1 Principle

An aliquot portion of stock solution (S1) or (S'1) (see ISO 10058-1:2008, Annex A) is transferred. Excess standard volumetric CyDTA solution is added to an aliquot portion of stock solution. A chelate compound of aluminium CyDTA is formed by adjusting the pH with ammonia water. The pH is further adjusted by addition of hexamethylenetetramine. The amount of remaining standard volumetric CyDTA is determined by back-titration with standard volumetric zinc solution using xylenol orange as an indicator. The content of aluminium oxide is calculated by allowing for the content of iron(III) oxide determined by the method given in Clause 5.

4.2 Procedure

4.2.1 Transfer precisely 50 ml of stock solution (S1), or stock solution (S'1), to a 300 ml beaker. Precisely add an adequate amount of standard volumetric CyDTA solution [$c(\text{CyDTA}) = 0,01 \text{ mol/l}$] and dilute to 100 ml with water.

The volume of standard volumetric CyDTA solution to be added depends on the percentage of aluminium oxide and iron(III) oxide as shown in Table 3.

Table 3 — Volumes of standard volumetric CyDTA solution

Mass fraction of aluminium oxide and iron(III) oxide %	Volume of standard volumetric CyDTA solution ml
< 1	5
≥ 1, < 2	10
≥ 2, < 4	20
≥ 4, < 7	30
≥ 7	40

4.2.2 Add 1 g of hexamethylenetetramine and a drop of methyl orange solution as an indicator. Using drops, add ammonia water (1+1) and ammonia water (1+9) of up to pH 3 until the solution indicates a slightly orange colour. Allow to stand for 5 min.

If ammonia water (1+9) is added excessively, the pH can be adjusted to less than 3, showing a red colour, by adding hydrochloric acid (1+1), before repeating the procedure in above paragraph.

4.2.3 Add 5 g of hexamethylenetetramine of up to pH 5,5 to pH 5,8, add 4 or 5 drops of xylenol orange solution as an indicator and titrate with standard volumetric zinc solution [$c(\text{Zn}) = 0,01 \text{ mol/l}$]. Carry out the titration gently while mixing. Record the end point of the titration when the colour changes from yellow to the first appearance of a permanent reddish colour.

4.3 Blank test

Using a 50 ml aliquot portion of blank solution (B1), or blank solution (B'1) (see ISO 10058-1:2008, Annex A), carry out the procedure in accordance with 4.2. Ensure that CyDTA standard volumetric solution is the same as that used for the corresponding stock solution.

4.4 Calculation

Calculate the mass fraction of aluminium oxide, $w_{\text{Al}_2\text{O}_3}$, expressed as a percentage, using Equation (3).

$$w_{\text{Al}_2\text{O}_3} = \frac{(V_2 - V_1) \times F \times 0,001\,019\,6}{m} \times \frac{250}{50} \times 100 - [(w_{\text{Fe}_2\text{O}_3} + w_{\text{TiO}_2}) \times 0,638 + w_{\text{MnO}} \times 0,719 + w_{\text{ZrO}_2} \times 0,414] \quad (3)$$

where

V_1 is the volume of standard volumetric zinc solution in 4.2.3, in millilitres (ml);

V_2 is the volume of standard volumetric zinc solution in 4.3, in millilitres (ml);

F is the factor of standard volumetric zinc solution;

m is the mass of the weighed sample in ISO 10058-1:2008, 8.2.2.3.1 or 8.2.3.3, in grams (g);

$w_{\text{Fe}_2\text{O}_3}$ is the mass fraction of iron(III) oxide determined in 5.5 or in ISO 10058-3:2008, 3.2.6, expressed as a percentage;

w_{TiO_2} is the mass fraction of titanium(IV) oxide determined in 6.5 or in ISO 10058-3:2008, 3.2.6, expressed as a percentage;

w_{MnO} is the mass fraction of manganese oxide determined in 7.5 or in ISO 10058-3:2008, 3.2.6 or 4.1.6, expressed as a percentage;

w_{ZrO_2} is the mass fraction of zirconium oxide determined in 13.5 or in ISO 10058-3:2008, 3.2.6, expressed as a percentage.

5 Determination of iron(III) oxide

5.1 Principle

Stock solution (S1) or (S'1) (see ISO 10058-1:2008, Annex A) is transferred and iron is reduced with L (+)-ascorbic acid. 1,10-Phenanthroline chloride is added, the pH is adjusted by adding ammonium acetate and the iron coloured complex with phenanthroline is developed. The absorbance is measured.

5.2 Procedure

5.2.1 Transfer an aliquot portion, determined in accordance with Table 4, of either stock solution (S1) or (S'1), to a 100 ml volumetric flask.

NOTE The aliquot portion volumes of stock solutions (S1) or (S'1) shown in Table 4 correspond to the content of iron(III) oxide in the sample.

Table 4 — Aliquot volumes of stock solution (S1) or (S'1)

Mass fraction of iron(III) oxide %	Aliquot volume of stock solution (S1) or (S'1) ml
< 0,5	25
≥ 0,5, < 1,5	10
≥ 1,5, < 5	5
≥ 5	2 ^a

^a Alternatively, transfer precisely 20 ml of the stock solution to a 100 ml volumetric flask and dilute to the mark with water. An aliquot portion of 10 ml may be used instead of the 2 ml of the stock solution.

5.2.2 Dilute to about 60 ml with water and, while mixing, add 5 ml of L (+)-tartaric acid solution and 2 ml of L (+)-ascorbic acid solution. Add 10 ml of 1,10-phenanthroline chloride solution and 10 ml of ammonium acetate solution. Dilute to the mark with water and allow to stand for 30 min. Measure the absorbance of the solution in a 10 mm cell at a wavelength of 510 nm against water.

5.3 Blank test

Using an aliquot portion of blank solution (B1) or blank solution (B'1) (see ISO 10058-1:2008, Annex A), carry out the procedure in accordance with 5.2. Use the same volume of aliquot portion for the blank solution as used for the corresponding stock solution.

5.4 Plotting of calibration graph

Transfer 0 ml (as reference), 5 ml, 10 ml and 15 ml aliquot portions [0 mg to 0,6 mg as iron(III) oxide] of the dilute standard iron(III) oxide solution (Fe_2O_3 0,04 mg/ml) to separate 100 ml volumetric flasks. Treat these solutions in accordance with 5.2.2 and measure the absorbance against the reference solution. Plot the relation between the absorbance measurements and mass of iron(III) oxide. Prepare the calibration graph by adjusting the curve so that it passes through the point of origin.

5.5 Calculation

Calculate the mass fraction of iron(III), $w_{\text{Fe}_2\text{O}_3}$, expressed as a percentage, using Equation (4). Use the amount of iron(III) oxide derived from the absorbance in 5.2 and 5.3, and the calibration in 5.4.

$$w_{\text{Fe}_2\text{O}_3} = \frac{m_s - m_b}{m} \times \frac{250}{V} \times 100 \quad (4)$$

where

m_s is the mass of iron(III) oxide in the aliquot portion of stock solution (S1) or (S'1), in grams (g);

m_b is the mass of iron(III) oxide in the aliquot portion of blank solution (B1) or (B'1), 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);

V is the volume of the aliquot portion taken from stock solution (S1) or (S'1) in 5.2.1, in millilitres (ml).

6 Determination of titanium(IV) oxide

6.1 Principle

Sample solution (S1) or (S'1) (see ISO 10058-1:2008, Annex A) is transferred. After the adjustment of acidity, iron is reduced with the addition of L (+)-ascorbic acid. The titanium is coloured by the di-antipyrylmethane (DAM) and the absorbance is measured.

6.2 Procedure

6.2.1 Transfer an aliquot portion, determined using Table 5, of either stock solution (S1) or (S'1) (see ISO 10058-1:2008, Annex A), to a 50 ml volumetric flask.

NOTE The aliquot portions taken from stock solution (S1) or (S'1) shown in Table 5 correspond to the content of titanium(IV) oxide in the sample.

Table 5 — Aliquot volumes taken from stock solution (S1) or (S'1)

Mass fraction of titanium(IV) oxide %	Aliquot volume taken of (S1) or (S'1) ml
< 0,5	25
≥ 0,5, < 1,5	10
≥ 1,5	5

6.2.2 Add 5 ml of hydrochloric acid (1+1), 2 ml of L (+)-ascorbic acid and allow to stand for 1 min. Add 15 ml of DAM solution, dilute to the mark with water and allow to stand for 90 min. Measure the absorbance of the solution in a 10 mm cell at a wavelength of 390 nm against water.

6.3 Blank test

Using the same aliquot portion of stock solution used in blank test solution (B1) or (B'1) (see ISO 10058-1:2008, Annex A), carry out the procedure in 6.2.