
**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**

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*Analyse chimique des produits de magnésite et de dolomie (méthode
alternative à la méthode par fluorescence de rayons X) —*

*Partie 1: Appareillage, réactifs, mise en solution et détermination de la
teneur en silice par gravimétrie*

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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 10058-1 was prepared by Technical Committee ISO/TC 33, *Refractories*.

This first edition of ISO 10058-1, together with ISO 10058-2 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 1: Apparatus, reagents, dissolution and determination of gravimetric silica

1 Scope

This part of ISO 10058 specifies apparatus, reagents, dissolution and gravimetric silica analysis for the chemical analysis of magnesite and dolomite refractory products and raw materials.

This part of ISO 10058 is applicable to the ranges of determination given in Table 1.

ISO 10058 specifies procedures for the chemical analysis of magnesite and dolomite refractory products and raw materials 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 6353-1, *Reagents for chemical analysis — Part 1: General test methods*

ISO 6353-2, *Reagents for chemical analysis — Part 2: Specifications — First series*

ISO 6353-3, *Reagents for chemical analysis — Part 3: Specifications — Second series*

ISO 26845:2008, *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 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 26845 apply.

4 Apparatus

Normal laboratory apparatus and other apparatus as defined in ISO 26845.

5 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

Reagents shall conform to the requirements of ISO 6353-1, ISO 6353-2 and ISO 6353-3 as appropriate. Specific requirements for reagents are given in the appropriate clause.

Use reagents that are defined in ISO 26845 and the following.

5.1 Standard volumetric solutions.

5.1.1 Standard volumetric CyDTA solution, $c(\text{CyDTA}) = 0,01 \text{ mol/l}$.

Add 8 ml of sodium hydroxide solution (100 g/l) and approximately 150 ml of water to 3,65 g of trans-1,2-cyclohexanediamine -*N,N,N',N'*-tetraacetic acid monohydrate (CyDTA), and dissolve by heating. After cooling, dilute to 1 000 ml with water; (approximately 25 ml of water are required).

Determine the exact strength of this solution by titration against the standard volumetric zinc solution, $c(\text{Zn}) = 0,01 \text{ mol/l}$.

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5.1.2 Standard volumetric zinc solution, $c(\text{Zn}) = 0,01 \text{ mol/l}$.

Wash the surface of the zinc (purity greater than 99,9 % by mass) with hydrochloric acid (1+3) and dissolve the oxidized layer; subsequently, wash with water, ethanol and diethyl ether in succession, then dry in a desiccator. Weigh 0,65 g (recorded to the nearest 0,000 1 g) of zinc and transfer it to a 300 ml beaker. Cover with a watch glass. Add 20 ml of water and 5 ml of nitric acid carefully, and heat to dissolve on a steam bath. After cooling, dilute to 1 000 ml in a volumetric flask with water.

Calculate the factor, F , of this zinc solution using Equation (1).

$$F = \frac{m}{0,6538} \times \frac{A}{100} \quad (1)$$

where

m is the mass of the weighed zinc, in grams (g);

A is the purity of the zinc, as a percentage by mass (%).

5.1.3 Standard volumetric EDTA solution, $c(\text{EDTA}) = 0,02 \text{ mol/l}$.

Dissolve 7,5 g of EDTA_2Na (ethylenediamine-tetraacetic acid disodium salt dihydrate) in 1 l of water. Store in a plastic bottle.

Determine the factor, F , of the 0,02 mol/l standard volumetric EDTA solution as follows.

Transfer 50 ml of 0,01 mol/l zinc solution, obtained in 5.1.2, to a 300 ml beaker and add 50 ml of water. Drop in sodium hydroxide solution (100 g/l) of up to pH 6 to pH 8 and add 2 ml of buffer solution (pH 10) and, while stirring, add 3 to 4 drops of Eriochrome Black T solution as an indicator and titrate with 0,02 mol/l standard volumetric EDTA solution. Observe and record the end point, which is when the reddish purple colour of the solution is altered to blue.

Calculate the factor, F , of the 0,02 mol/l standard volumetric EDTA solution using Equation (2).

$$F = \frac{f \times 50}{2 \times V} \quad (2)$$

where

f is the factor of 0,01 mol/l standard volumetric zinc solution;

V is the volume of 0,02 mol/l standard volumetric EDTA solution, in millilitres (ml).

5.2 Standard solutions.

5.2.1 Standard aluminium oxide solution, Al_2O_3 1 mg/ml.

Wash the surface of a sufficient amount of aluminium metal (approximately 0,6 g to 0,7 g), purity of more than 99,9 % by mass, with hydrochloric acid (1+4) to dissolve the oxidized layer. Then wash with water, ethanol and diethyl ether in succession, and dry in a desiccator. Weigh 0,529 2 g of aluminium and transfer it to a 250 ml beaker. Cover the beaker with a watch glass, add 20 ml hydrochloric acid (1+1), and heat to dissolve. After cooling, dilute to 1 000 ml with water in a volumetric flask.

5.2.2 Standard calcium oxide solution, CaO 1 mg/ml.

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Dissolve 1,785 g of pure calcium carbonate that has been previously dried at 150 °C for 2 h, in a slight excess of diluted hydrochloric acid (1+4) in a 250 ml beaker, covered with a watch glass. Boil to expel carbon dioxide, cool and dilute to 1 000 ml with water in a volumetric flask.

5.2.3 Standard chromium(III) oxide solution, Cr_2O_3 1 mg/ml.

Dry between 2 g to 3 g of potassium dichromate at 110 °C for at least 2 h. Weigh 1,935 5 g of this and dissolve in water. Dilute to 1 000 ml with water in a volumetric flask.

5.2.4 Dilute standard chromium(III) oxide solution, Cr_2O_3 0,025 mg/ml.

Pipette 25 ml of the standard chromic oxide solution (1 mg/ml) to a 1 000 ml volumetric flask and dilute to the mark with water. Prepare this solution freshly when required.

5.2.5 Standard iron(III) oxide solution, Fe_2O_3 1 mg/ml.

Wash the surface of a sufficient amount of iron metal (purity greater than 99,9 % by mass) with hydrochloric acid (1+4). Then dissolve the oxidized layer, wash with water, ethanol, and diethyl ether in succession. Dry in a desiccator. Weigh 0,699 4 g of this, transfer to a beaker (200 ml) and cover with a watch glass. Add 40 ml of hydrochloric acid (1+1) and heat on a steam bath until dissolved. After cooling, dilute to 1 000 ml with water in a volumetric flask.

5.2.6 Dilute standard iron(III) oxide solution, Fe_2O_3 0,2 mg/ml.

Pipette 200 ml of the standard iron(III) oxide solution (Fe_2O_3 1 mg/ml) into a 1 000 ml volumetric flask and dilute to the mark with water. Prepare this solution freshly when required.

5.2.7 Dilute standard iron(III) oxide solution, Fe₂O₃ 0,04 mg/ml.

Pipette 40 ml of the standard iron(III) oxide solution (Fe₂O₃ 1 mg/ml) into a 1 000 ml volumetric flask and dilute to the mark with water. Prepare this solution freshly when required.

5.2.8 Standard magnesium oxide solution, MgO 1 mg/ml.

Wash the surface of a sufficient amount of magnesium metal (purity greater than 99,9 % by mass) with hydrochloric acid (1+1) to dissolve the oxidized layer. Wash with water, ethanol and diethyl ether in succession and dry in a desiccator. Weigh 0,301 5 g of the washed magnesium, transfer to a 200 ml beaker and cover with a watch glass. Add 20 ml of hydrochloric acid (1+1), and heat on a steam bath until dissolved. After cooling, transfer to a 500 ml volumetric flask, and dilute to the mark with water.

5.2.9 Standard manganese(II) oxide solution, MnO 1 mg/ml.

Wash the surface of a sufficient mass of manganese metal (purity greater than 99,9 % by mass) with hydrochloric acid (1+3) to dissolve the oxidized layer. Then wash with water, ethanol, and diethyl ether in succession and dry in a desiccator. Weigh 0,774 5 g of this metal, transfer to a 200 ml beaker and cover with a watch glass. Add 40 ml of nitric acid (1+1) and heat to dissolve; after cooling, transfer to a 1 000 ml volumetric flask.

5.2.10 Dilute standard manganese(II) oxide solution, MnO 0,04 mg/ml.

Transfer 40 ml of the standard manganese(II) oxide solution (MnO 1 mg/ml) to a 1 000 ml volumetric flask and dilute to the mark with water. Prepare this solution freshly as required.

5.2.11 Standard phosphorus(V) oxide solution, P₂O₅ 1 mg/ml.

Heat about 3 g of potassium dihydrogen phosphate at 110 °C ± 5 °C for 3 h, and allow to cool in a desiccator. Weigh 1,917 6 g, transfer to a beaker and dissolve with approximately 300 ml of water. Dilute to 1 000 ml with water in a volumetric flask. <https://standards.iteh.ai/catalog/standards/sist/dd5d05b4-5275-4764-b6f1-2239acd65fed/iso-10058-1-2008>

5.2.12 Dilute standard phosphorus(V) oxide solution, P₂O₅ 0,04 mg/ml.

Transfer precisely 40 ml of the standard phosphorus(V) oxide solution (5.2.11) into a 1 000 ml volumetric flask and dilute to the mark with water.

5.2.13 Dilute standard phosphorus(V) oxide solution, P₂O₅ 0,01 mg/ml.

Pipette 10 ml of the standard phosphorus(V) oxide solution (5.2.11) into a 1 000 ml volumetric flask and dilute to the mark with water. Prepare this solution freshly when required.

5.2.14 Standard potassium oxide solution, K₂O 1 mg/ml.

Transfer 1 g to 1,5 g of potassium chloride into a platinum crucible (e.g. 30 ml) and ignite at 600 °C ± 25 °C for approximately 60 min. Allow the crucible and contents to cool in a desiccator. Weigh 0,791 4 g of this and transfer to a 200 ml beaker. Dissolve in 100 ml of water, transfer to a 500 ml volumetric flask, and dilute to the mark with water.

5.2.15 Standard silicon(IV) oxide solution, SiO₂ 1 mg/ml.

Weigh 1,5 g to 2 g of silicon(IV) oxide (purity greater than 99,9 % by mass) in a platinum crucible (e.g. 30 ml) and heat for 30 min at 1 150 °C ± 50 °C. Cool in a desiccator and then weigh 1,000 g of this silicon(IV) oxide into a platinum crucible (e.g. 50 ml). Fuse the silicon(IV) oxide with 5,0 g of anhydrous sodium carbonate. Cool and wipe the outside of the crucible, and dissolve in warm water (150 ml) in a plastic 200 ml beaker while stirring using a plastic rod. Cool and dilute without heating to 1 000 ml in a volumetric flask. Transfer this solution to a plastics bottle immediately.

5.2.16 Dilute standard silicon(IV) oxide solution, SiO₂ 0,08 mg/ml.

Pipette 40 ml of the standard silicon(IV) oxide solution (5.2.15) into a 500 ml volumetric flask and dilute to the mark with water. Prepare this solution freshly when required.

5.2.17 Dilute standard silicon(IV) oxide solution, SiO₂ 0,04 mg/ml.

Pipette 20 ml of the standard silicon(IV) oxide solution (5.2.15) into a 500 ml volumetric flask and dilute to the mark with water. Prepare this solution freshly when required.

5.2.18 Standard sodium oxide solution, Na₂O 1 mg/ml.

Transfer 1 g to 1,5 g of sodium chloride into a platinum crucible (e.g. 30 ml) and ignite at 600 °C ± 25 °C for approximately 60 min. Allow the crucible and contents to cool in a desiccator. Weigh 0,942 9 g of this and transfer to a 200 ml beaker. Dissolve in 100 ml of water, transfer to a 500 ml volumetric flask, and dilute to the mark with water.

5.2.19 Standard titanium(IV) oxide solution, TiO₂ 1 mg/ml.

Wash the surface of a sufficient amount of titanium metal (purity greater than 99,9 % by mass) with hydrochloric acid (1+3) and dissolve the oxidized layer. Subsequently, wash with water, ethanol and diethyl ether in succession, then dry in a desiccator. Weigh 0,599 4 g of this titanium metal, and transfer to a platinum dish. Cover with a watch glass made of ethylene 4-fluoride resin, then add 40 ml of hydrofluoric, 15 ml of sulfuric (1+1) and 2 ml nitric acid, and heat to dissolve on a steam bath. Remove the watch glass, rinse the watch glass with water and heat the solution on a sand bath until the appearance of strong sulfuric acid fumes. After cooling, rinse the inner wall of the platinum dish with a small amount of water and heat again until fumes are seen. After cooling, dilute to 1 000 ml with water in a volumetric flask.

5.2.20 Dilute standard titanium(IV) oxide solution, TiO₂ 0,01 mg/ml.

Pipette 10 ml of the standard titanium(IV) oxide solution (5.2.19) into a 1 000 ml volumetric flask and dilute to the mark with water. Prepare this solution freshly when required.

5.2.21 Standard zirconium oxide solution, ZrO₂ 1 mg/ml.

Transfer about 0,3 g of zirconium oxide (purity greater than 99,9 % by mass) into a platinum crucible (e.g. 30 ml), heat strongly at 1 150 °C ± 50 °C for approximately 30 min, and allow to cool in a desiccator. Weigh 0,200 0 g of this into a platinum crucible (e.g. 30 ml), add 4 g of potassium disulfate and fuse, covering with a platinum lid.

Fuse over a gas burner at as low a temperature as possible, otherwise sulfur trioxide will be lost before the attack is complete.

After cooling, put the platinum crucible into a 200 ml beaker, add 100 ml of sulfuric acid (1+9), and warm to dissolve. After cooling, remove and wash the platinum crucible with water, transfer to a 200 ml volumetric flask and dilute to the mark with water.

5.2.22 Dilute standard zirconium oxide solution, ZrO₂ 0,005 mg/ml.

Pipette 5 ml of the standard zirconium oxide solution (5.2.21) into a 1 000 ml volumetric flask and dilute to the mark with water. Prepare this solution freshly when required.

5.2.23 Standard scandium solution, Sc 1,0 mg/ml.

Dry about 2,0 g of scandium oxide at 110 °C ± 5 °C for 60 min and cool in a desiccator. Weigh 1,533 8 g of this and transfer it to a 600 ml beaker. Gradually add 100 ml of hydrochloric acid (1+1) to dissolve and dilute precisely to 1 000 ml with water in a volumetric flask.

A suitable commercial standard solution may be used.