



SLOVENSKI STANDARD SIST EN ISO 10058:1998

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Magneziti in dolomiti - Kemijska analiza (ISO 10058:1992)

Magnesites and dolomites - Chemical analysis (ISO 10058:1992)

Magnesit und Dolomit - Chemische Analyse (ISO 10058:1992)

Produits de magnésie et de dolomie - Analyse chimique (ISO 10058:1992)

Ta slovenski standard je istoveten z: **EN ISO 10058:1996**

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January 1996

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English version

**Magnesites and dolomites - Chemical analysis
(ISO 10058:1992)**Produits de magnésie et de dolomie - Analyse
chimique (ISO 10058:1992)Magnesit und Dolomit - Chemische Analyse
(ISO 10058:1992)**iTeh STANDARD PREVIEW
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Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

Foreword

The text of the International Standard from ISO/TC 33 "Refractories" of the International Organization for Standardization (ISO) has been taken over as a European Standard by the Technical Committee CEN/TC 187 "Refractory products and materials".

This European Standard shall be given the status of a National Standard, either by publication of an identical text or by endorsement, at the latest by July 1996, and conflicting national standards shall be withdrawn at the latest by July 1996.

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The text of the International Standard ISO 10058:1992 has been approved by CEN as a European Standard without any modification.

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INTERNATIONAL STANDARD

ISO
10058

First edition
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Magnesites and dolomites — Chemical analysis

Produits de magnésie et de dolomie — Analyse chimique

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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.

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.

International Standard ISO 10058 was prepared by Technical Committee ISO/TC 33, *Refractories*, Sub-Committee SC 2, *Methods of testing*.

Annex A forms an integral part of this International Standard.

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Magnesites and dolomites — Chemical analysis

1 Scope

This International Standard specifies methods for the determination of silica, alumina, titania, iron oxide and oxides of manganese, chromium, calcium, magnesium, sodium, potassium and lithium. It also specifies methods for determining the loss on ignition of magnesite and dolomite, and of refractories based on these raw materials.

Annex A describes a method for the determination of the boron content of magnesites only.

NOTE 1 Physical methods are used increasingly for chemical analysis. At present, it is not possible to describe a standardized test method, because the type of apparatus used is important.

2 Dissolution and determination of silica

2.1 Principle

Decompose the sample with hydrochloric acid and separate the silica by coagulation with a polyethylene oxide solution. Filter, wash the residue, heat and weigh it, and submit it to a treatment with hydrofluoric and sulfuric acids. After this treatment, weigh the remaining residue again and fuse it in sodium carbonate and boric acid. It is then dissolved in the filtrate from the silica, and the solution is diluted to a standard volume to obtain the stock solution (A) of the sample.

In an aliquot, the small quantity of silica not separated by coagulation is subsequently determined by a spectrophotometric method based on the formation of molybdenum blue, using alternatively iron(II) sulfate or tin(II) chloride as a reducing agent. The absorbance maximum of the reduced silicomolybdate complex lies at a wavelength of 810 nm.

2.2 Reagents

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

2.2.1 Sodium carbonate, anhydrous.

2.2.2 Boric acid, powdered.

2.2.3 Polyethylene oxide solution, 2,5 g/l.

Add 0,5 g of polyethylene oxide to 200 ml of water while stirring slowly, preferably with a mechanical stirrer, until dissolved. Discard after 2 weeks.

2.2.4 Accelerator granules, ashless, of mass about 1 g.

2.2.5 Hydrochloric acid, concentrated, $\rho = 1,19$ g/ml.

2.2.6 Hydrochloric acid, diluted 1 + 19.

Add 1 volume of hydrochloric acid (2.2.5) to 19 volumes of water.

2.2.7 Sulfuric acid, concentrated, $\rho = 1,84$ g/ml.

2.2.8 Hydrofluoric acid, 40 % (m/m).

2.3 Apparatus

Usual laboratory apparatus and the following.

2.3.1 Sand bath or hot plate.

2.3.2 Muffle furnace, capable of being controlled at 1 180 °C to 1 200 °C.

2.3.3 Platinum crucible.

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2.4 Procedure

Weigh into a 250 ml beaker, 5,000 g¹⁾ of finely ground analytical sample dried at 110 °C. Add 25 ml of water and 40 ml of concentrated hydrochloric acid (2.2.5) and cover with a watch-glass. Transfer to a sand bath or a hot-plate (2.3.1) and boil for 30 min. Allow the beaker and contents to cool, rinse the watch-glass with water, add the accelerator granules (2.2.4) and stir to break up the pulp. Then add, stirring all the time, 5 ml of polyethylene oxide solution (2.2.3) and allow to stand for 5 min. Filter the solution through a closed-pore paper filter suitable for separating very fine precipitates. Transfer the residue (containing the precipitate of silica) quantitatively with hot diluted hydrochloric acid (2.2.6) to the filter paper. Wash the precipitate six times with hot diluted hydrochloric acid (2.2.6) and then with hot water until free from chlorides (up to a volume of about 400 ml). Store the filtrate. Transfer the filter paper and precipitate to a heated and weighed platinum crucible (2.2.3). Heat at a low temperature until the precipitate is free from carbonaceous matter, then heat it in the muffle furnace (2.3.2) controlled at 1 180 °C to 1 200 °C to constant mass (m_1), a time of 15 min normally being sufficient.

Moisten the residue in the cold crucible with water, add 5 drops of concentrated sulfuric acid (2.2.7) and 10 ml of hydrofluoric acid (2.2.8). Evaporate to dryness in the sand bath or on the hot-plate in a fume cupboard.

Heat the crucible and the residue, cautiously at first over a Bunsen burner, and finally for 5 min between 1 180 °C and 1 200 °C in the muffle furnace. Allow to cool in a desiccator and weigh (mass m_2). If the residue weighs more than 30 mg, repeat the treatment with hydrofluoric and sulfuric acids to ensure that all the silica is removed. The difference between the two masses ($m_1 - m_2$) represents the "gravimetric" silica.

Fuse the residue from the hydrofluoric and sulfuric acid treatment with 2 g of the anhydrous powdered sodium carbonate (2.2.1) and 0,7 g of powdered boric acid (2.2.2). Allow the melt to cool and dissolve in the filtrate from the main silica. Cool, transfer the solution to a 500 ml one-mark volumetric flask, dilute to volume with water and mix.

This solution will become stock solution A of the sample for the spectrophotometric determination of the residual silica in accordance with 2.5.1 or 2.5.2 and for the determination of alumina (clause 3), iron(III) oxide (clause 4), titanium(IV) oxide (clause 5), manganese(IV) oxide (clause 6), chromium(III) oxide (clause 7), calcium oxide (clause 8) and magnesium oxide (clause 9).

2.5 Spectrophotometric determination of residual silica in filtrate

2.5.1 Reduction to molybdenum blue complex with iron(II) sulfate

2.5.1.1 Reagents

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

2.5.1.1.1 Ammonium hydroxide solution,

$\rho = 0,9\text{g/ml}$.

Although the method allows for the presence of silica in the ammonium hydroxide solution, the reagent should contain as little silica as possible. It should be noted that ammonium hydroxide solution stored in glass bottles will dissolve silica from the glass.

2.5.1.1.2 Ammonium iron(III) sulfate solution.

Dissolve 100 g of ammonium iron(III) sulfate dodecahydrate $[\text{NH}_4\text{Fe}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}]$ in water, add 300 ml of concentrated hydrochloric acid (2.2.5) and dilute to 1 litre with water.

2.5.1.1.3 Ammonium molybdate solution.

Dissolve 80 g of hexammonium heptamolybdate tetrahydrate $[(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}]$ in water, filter if necessary, dilute to 1 litre and mix. Store in a polyethylene bottle. Renew the solution after 4 weeks, or earlier if any appreciable deposit is observed.

2.5.1.1.4 Oxalic acid solution.

Dissolve 100 g of oxalic acid dihydrate ($\text{C}_2\text{H}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$) with water and dilute to 1 litre.

2.5.1.1.5 Iron(II) sulfate solution.

Dissolve 10 g of iron(II) sulfate heptahydrate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) in water, add 4 drops of diluted sulfuric acid (1 + 1) and dilute to 100 ml with water. Prepare this solution freshly before use.

2.5.1.1.6 Silica stock solution, containing 0,5 g of SiO_2 per litre.

Ignite a sample of high purity silica to 1 000 °C and cool. Then weigh 0,250 g of this sample in a platinum crucible with 5 g of anhydrous sodium carbonate. Dissolve the melt in a polyethylene vessel in 300 ml of water while adding 20 g of sodium hydroxide.

1) In the case of natural iron and silica-rich magnesite, the sample mass may optionally be reduced to 2,000 g. The aliquots to be taken for the determination are to be changed accordingly.

Cool the solution to 20 °C, transfer to a 500 ml one-mark volumetric flask and make up to the mark with water. Stir thoroughly and transfer to a polyethylene flask for storage. This solution will remain stable for 6 months.

1 ml of this standard solution contains 0,5 mg of SiO₂.

2.5.1.1.7 Silica standard solution, containing 0,02 g of SiO₂ per litre.

Dilute 20 ml of the silica stock solution (2.5.1.1.6) to 500 ml with water in a one-mark volumetric flask. Prepare this solution daily if required.

1 ml of this standard solution contains 0,02 mg of SiO₂.

2.5.1.1.8 Phenolphthalein solution.

Dissolve 100 g of phenolphthalein in ethanol and dilute to 100 ml.

2.5.1.2 Apparatus

Usual laboratory apparatus and a spectrophotometer, fitted with cells of an appropriate size.

2.5.1.3 Procedure

2.5.1.3.1 Pipette two 5 ml aliquots of the stock solution A, prepared as in 2.4 into two 100 ml one-mark volumetric flasks. Add one drop of phenolphthalein solution (2.5.1.1.8) to each flask, and while continually shaking, add ammonium hydroxide solution (2.5.1.1.1) one drop at a time. As soon as the alkaline reaction produces a red colour, immediately add 5 ml of ammonium iron(III) sulfate solution (2.5.1.1.2). To the first of the two solutions, add 5 ml of ammonium molybdate solution (2.5.1.1.3) and leave to stand for 20 min.

NOTE 2 Cloudiness may occur but it does not cause any interference and disappears after the addition of oxalic acid.

Add 20 ml of oxalic acid solution (2.5.1.1.4) and shake the solution until it is clear. Add 1 ml of iron(II) sulfate solution and make up to the mark with water. Take the second 5 ml aliquot part of the stock solution A (see 2.4) which will serve as a reference, add 20 ml of oxalic acid solution to it and leave it also to stand for 20 min. Add 5 ml of ammonium molybdate solution, then 1 ml of iron(II) sulfate solution and make up to the mark with water. Carry out a blank determination on a solution containing all the reagents, but omitting the stock solution (see 2.4).

2.5.1.3.2 Measure, in cells of an appropriate size, the absorbance of the solution brought to 20 °C ± 2 °C, using the optimum wavelength of 810 nm, and compare it with the reference solution

(2.5.1.3.1). Subtract the absorbance of the blank solution from that of the sample solution.

2.5.1.3.3 The blank solution is treated and measured in the same manner as the sample. Subtract the absorbance of the blank solution from that of the sample solution.

2.5.1.4 Plotting the calibration graph.

Pipette 0 ml, 2 ml, 4 ml, 6 ml, 8 ml and 10 ml volumes of the silica standard solution (2.5.1.1.7) into six 100 ml one-mark volumetric flasks.

Add one drop of the phenolphthalein solution (2.5.1.1.8) to the contents of each flask and while stirring constantly, mix with hydrochloric acid (1 + 4) until the red colour disappears. Then add the ammonium hydroxide solution (2.5.1.1.1) drop by drop. As soon as the alkaline reaction produces a red colour, immediately mix with 5 ml of the ammonium iron(III) sulfate solution (2.5.1.1.2) and 5 ml of the ammonium molybdate solution (2.5.1.1.3), and allow to stand for 20 min. Add 20 ml of the oxalic acid solution (2.5.1.1.4) and shake the solution until it becomes clear. Add 1 ml of the iron(II) sulfate solution (2.5.1.1.5) and make up to the mark with water.

Measure the absorbance of each calibration solution maintained at 20 °C ± 2 °C using the spectrophotometer set at an optimum wavelength of 810 nm against the zero member of the set of calibration solutions as a reference.

Use the values thus obtained to plot a calibration graph. On the basis of this graph, determine the mass of silica contained in 5 ml of the stock solution A.

2.5.1.5 Expression of results

The silica content, $w(\text{SiO}_2)$, expressed as a percentage by mass, is given by the following formula:

$$w(\text{SiO}_2) = \frac{(m_1 - m_2) + (m_3 - m_4)}{m_0} \times 100$$

where

- m_0 is the mass, in grams, of the test portion;
- m_1 is the mass, in grams, of the crucible and its contents before treatment with hydrofluoric and sulfuric acids;
- m_2 is the mass, in grams, of the crucible with the residue after treatment with hydrofluoric and sulfuric acids;
- m_3 is the mass, in grams, of silica contained in 5 ml of the stock solution A;