
रासायनिक उद्योग के लिए मैग्नेसाइट —
विशिष्टि
(दूसरा पुनरीक्षण)

Magnesite for Chemical Industry —
Specification
(Second Revision)

ICS 71.060.50

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FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Inorganic Chemicals Sectional Committee had been approved by the Chemical Division Council.

This standard was first published in 1966 and subsequently revised in 1979. In the first revision, the limit of iron oxide was lowered from 1.0 to 0.4. Changes were also made in the method of test for calcium.

In this revision, instrumental test methods for the determination of calcium and magnesium have been added as alternate test methods. In addition to this, editorial corrections have been made wherever required. Also, reference clause has been incorporated. Further, packing and marking clause has been updated.

Magnesite occurs in two forms, namely, crystalline and amorphous. Amorphous form is commonly found in India. It is white in colour and breaks with conchoidal fracture. Magnesite is used in the manufacture of dead burnt magnesium oxide, epsom salts and caustic magnesia for magnesium oxychloride compositions, and in rubber and other chemical industries.

The composition of the Committee responsible for formulation of this standard is given in [Annex D](#).

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Indian Standard***MAGNESITE FOR CHEMICAL INDUSTRY — SPECIFICATION***(Second Revision)***1 SCOPE**

This standard prescribes the requirements and the methods of sampling and test for magnesite used in chemical industry.

2 REFERENCES

The standards listed in [Annex A](#) contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this Indian Standard are encouraged to investigate the possibility of applying the most recent edition of these standards.

3 REQUIREMENTS**3.1 Description**

The material shall be in the form of white lumps, and free from dirt and other foreign matter.

3.2 The material shall comply with the requirements given in [Table 1](#), when tested according to the methods prescribed in [Annex B](#) of this standard and IS 1760 (Part 1), (Part 2) and (Part 3). Reference to the relevant test methods is given in col (4) and (5) of the table.

4 PACKING AND MARKING**4.1 Packing**

Unless otherwise agreed between the purchaser and the supplier, the material shall be supplied in open wagons.

4.2 Marking

A good sized metallic or cardboard label bearing the following information with suitable paint or ink shall be conspicuously displayed on the carrier and also placed inside:

a) Name of the material;

- b) Name of the supplier and the recognized trade-mark, if any;
- c) Gross and net mass;
- d) Date of supply; and
- e) Batch number.

4.2.1 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

5 SAMPLING**5.1 Preparation of Test Samples**

Representative samples of the material shall be drawn as prescribed in IS 2109.

5.2 Number of Tests

Tests for the determination of magnesium, silica and loss on ignition shall be conducted on each of the individual samples. Tests for the determination of the remaining characteristics shall be performed on the composite sample.

5.3 Criteria for Conformity

The lot shall be declared as conforming to the requirements of this specification if the conditions given in [5.3.1](#) and [5.3.2](#) are satisfied.

5.3.1 For each of the characteristics tested on individual samples (*see* [5.2](#)), all the test results satisfy the relevant requirements given in the specification.

5.3.2 All the test results on the composite sample satisfy relevant requirements given in the specification.

Table 1 Requirements for Magnesite for Chemical Industry*(Clause [3.2](#))*

Sl No.	Characteristic	Requirement	Method of Tests, Ref to	Method of Tests, Ref to
(1)	(2)	(3)	(4)	(5)
i)	Loss on ignition, percent by mass, <i>Min</i>	48.0	–	IS 1760 (Part 1)
ii)	Silica (as SiO ₂), percent by mass, <i>Max</i>	2.0	–	IS 1760 (Part 2)
iii)	Alumina (as Al ₂ O ₃), percent by mass <i>Max</i>	0.3	–	IS 1760 (Part 3)
iv)	Iron oxide (as Fe ₂ O ₃), percent by mass, <i>Max</i>	0.4	–	IS 1760 (Part 3)
v)	Magnesium (MgO), percent by mass, <i>Min</i>	45.0	Annex C	IS 1760 (Part 3)
vi)	Calcium (as CaO), percent by mass, <i>Max</i>	1.0	Annex B or Annex C	–

ANNEX A

(Clause 2)

LIST OF REFERRED STANDARDS

<i>IS No.</i>	<i>Title</i>	<i>IS No.</i>	<i>Title</i>
IS 264 : 2005	Nitric acid — Specification (<i>third revision</i>)	IS 2109 : 1982	Methods of sampling dolomite, limestone and other allied materials (<i>first revision</i>)
IS 265 : 2021	Hydrochloric acid — Specification (<i>fifth revision</i>)	IS 3025	Methods of sampling and test (physical and chemical) for water and wastewater:
IS 1070 : 2023	Reagent grade water — Specification (<i>fourth revision</i>)	(Part 2) : 2019/ ISO 11885 : 2007	Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES) (<i>first revision</i>)
IS 1760	Chemical analysis of limestone, dolomite and allied materials:	(Part 65) : 2022/ ISO 17294-2 : 2016	Application of inductively coupled plasma mass spectrometry (ICP-MS) — Determination of selected elements including uranium isotopes (<i>first revision</i>)
(Part 1) : 1991	Determination of loss on ignition (<i>first revision</i>)		
(Part 2) : 1991	Determination of silica (<i>first revision</i>)		
(Part 3) : 1992	Determination of iron oxide, alumina, calcium oxide and magnesia (<i>first revision</i>)		

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ANNEX B

[Clause 3.2 and Table 1, Sl No.(vi)]

DETERMINATION OF CALCIUM IN MAGNESITE

B-1 GENERAL

B-1.1 Calcium may be determined by two methods, namely, EDTA titration method and phosphate method. For routine analysis, the EDTA method prescribed here shall be followed; in case of dispute, phosphate method as prescribed IS 1760 (Part 3) shall be followed.

B-1.2 Outline of the Method

The sample is treated with hydrochloric acid and insoluble matter is removed by filtration. Triethanol amine is added to form a complex with the R_2O_3 group and a known volume of standard EDTA is added. This volume is sufficient to complex all the calcium and the excess complexes its equivalent amount of magnesium; the remainder of the magnesium is then precipitated with sodium hydroxide. The magnesium EDTA complex is titrated with standard calcium solution using Patton and Reeder's indicator.

B-2 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

B-3 REAGENTS

B-3.1 Concentrated Hydrochloric Acid — *see* IS 265

B-3.2 Magnesium Sulphate Solution

Dissolve 25 g of magnesium sulphate ($MgSO_4 \cdot 7H_2O$) in water, filter and dilute to 500 ml.

B-3.3 Sodium Hydroxide Solution — approximately 4 N

B-3.4 Concentrated Nitric Acid — *see* IS 264

B-3.5 Triethanol Amine — 1 : 1 (v/v)

B-3.6 Standard Calcium Chloride Solution — 0.05 M

Dissolve 5.005 g of dried calcium carbonate in a slight excess of dilute hydrochloric acid (1 : 3). Boil to expel carbon dioxide, cool and dilute to 1 litre.

B-3.7 Standard Ethylenediamine Tetra-acetate (EDTA) Solution

Approximately 0.05 M. Dissolve 18.65 g of disodium tetra-acetate dihydrate in warm water, filter, cool and dilute to 1 litre.

B-3.8 Patton and Reeder's Indicator

Intimately mix by grinding, about 0.1 g of Patton and Reeder's indicator with about 50 g of anhydrous potassium sulphate. Store in a well-stoppered bottle protected from light.

B-3.9 Ascorbic Acid — solid**B-4 PROCEDURE****B-4.1 Standardization of EDTA Solution**

Pipette 25 ml of the EDTA solution into a 500 ml conical flask and add 50 ml of magnesium sulphate solution, dilute to about 250 ml and shake well. Then add 25 ml of sodium hydroxide solution followed by 0.25 g of the indicator and again shake well. Immediately titrate with the calcium chloride solution till colour changes from blue to wine red. EDTA solution shall be standardized only when required.

B-4.2 Weigh accurately about 1 g of the dried and finely ground sample into a 250 ml beaker. Add 5 ml of water and 10 ml of concentrated hydrochloric acid, cover the beaker with a watch-glass and boil until all soluble matter dissolves. Add a drop of nitric acid and boil off the fumes of nitrous oxide. Rinse any deposit from the underside of the watch-glass into the beaker. Dilute to about 100 ml, filter and wash, collecting the filtrate and washings in a 500 ml conical flask. Cool, add 5 ml of triethanolamine, and pipette into the solution 25 ml of EDTA solution, then add sodium hydroxide solution, drop by drop with shaking until a slight permanent precipitate remains. Dilute to about 250 ml, add 25 ml of the sodium hydroxide solution, and add about 1 g of the ascorbic acid and about 0.2 g of the indicator. Titrate immediately with the standard calcium chloride solution exactly as given in [B-4.1](#).

B-5 CALCULATION

If 25 ml of the EDTA solution is equivalent to *a* ml of calcium chloride solution and *b* ml of the latter solution is used in the back titration, then

percentage of calcium oxide in the dry unignited sample

$$= 0.2804 \times \frac{a-b}{m}$$

where

m = mass, in g, of the sample taken;

a = volume, in ml of calcium chloride solution;
and

b = volume, in ml, of calcium chloride solution used in back titration.

B-6 ALTERNATIVE METHOD

Calcium and magnesium may alternatively be determined by ICP-OES instrumental test method as prescribed at [Annex C](#) or ICP-MS method as prescribed in IS 3025 (Part 65). Instrumental test method using ICP-OES or ICP-MS shall be used as referee method in case of any dispute.

ANNEX C

([Table 1](#) and [B-6](#))

DETERMINATION OF CALCIUM AND MAGNESIUM BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD

C-1 PRINCIPLE

The sample solution under analysis is nebulized through a nebulizer inside a spray chamber. The aerosol formed is aspirated to argon plasma torch [produced by a radio-frequency inductively coupled plasma (ICP)], where the molecules break into constituent atoms and/or molecular species and atoms are get excited. These excited atoms then return back to the lower energy state by emitting radiation of specific wavelength. These emitted radiations are characteristic of an element and are measured by the Photomultiplier tube detector and intensity of such emitted radiation is directly proportional to the concentration of respective constituent element in the sample.

C-2 RECOMMENDED WAVELENGTH, LIMIT OF QUANTIFICATION AND IMPORTANT SPECTRAL INTERFERENCES

Elements along with the recommended wavelengths and typical estimated limits of quantification are listed in [Table 2](#). Actual working detection limits are dependent on the type of instrumentation, detection device and sample introduction system used and on the sample matrix. Therefore, these concentrations can vary between different instruments.

Additionally, [Table 2](#) lists the most important spectral interferences at the recommended wavelengths for analysis.

C-3 REAGENTS AND SOLUTIONS

C-3.1 Nitric Acid (65 Percent) Suprapure

C-3.2 Standard Stock Solution

Either prepare by dissolving proportionate amount of soluble compounds of elements (preferably spectroscopic grade), or use commercially available

certified stock solution of 10 µg/ml, 100 µg/ml or 1 000 µg/ml of lead, iron, calcium, magnesium, manganese, arsenic, molybdenum, aluminium and mercury in 2 percent to 5 percent nitric acid. It is preferable to prepare single stock solution of multi elemental standards for analysis.

C-3.3 Standard Solution

Pipette out 5 ml from 100 µg/ml standard stock solution into a 100 ml volumetric flask and make up volume with 2 percent nitric acid to prepare 5 µg/ml solution. From this 5 µg/ml solution, an aliquot of 1.0 ml, 3.0 ml and 5.0 ml taken in 50 ml volumetric flasks (separate) and make up volume with 2 percent nitric acid to prepare 0.1 µg/ml, 0.3 µg/ml and 0.5 µg/ml solution of respective elements under reference.

C-3.4 Sample Preparation

Weigh about 2.5 g of the sample in a 50 ml volumetric flask and add 1.0 ml nitric acid and make up the volume with water.

NOTE — Sample should be clear before injecting to the instrument.

C-3.5 Reagent Blank Solution

Place 50 ml of nitric acid and 1 000 ml of water into an HDPE or PP container. For ultra-trace analysis, polytetrafluoroethylene (PTFE) containers should be used. Prior to analysis, make sure that the acid matrix and concentration of the reagent blank solution is the same as in the standard and sample solutions.

C-4 INSTRUMENT

Set up the instrument as per the manufacturer's instructions manual for recommended operating parameters, based on the manufacturers operating

manual and evaluated by internal check analysis using of standard solution of element as well as data selected carefully from [Table 2](#).

For analysis of mercury and arsenic, a gas (hydride)/vapour generating system is coupled with ICP, instead of using of nebulizer. The mercury vapour/arsine generated through the system is carried by the carrier gas (Ar) to plasma torch, and other instrumental conditions shall be the same as above.

NOTE — Sensitivity, instrumental detection limit, precision, linear dynamic range and interference effects will be investigated and established for each individual analyte line on that particular instrument.

C-5 PROCEDURE

C-5.1 Calibration

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions ([C-3.5](#)). The relationship between concentration and intensity is linear up to six orders of magnitude. Examine the spectra of the element and make necessary adjustments (if required) for the exact peak positions and baselines to ensure proper measurements of the respective peak intensities. Flush the system with the reagent blank solution between each standard.

C-5.2 Before beginning the sample run, re-analyse the reference standard with the highest concentration as if it were a sample. Ensure that the

concentration values do not deviate from the actual values by more than ± 5 percent (or the established control limits, whichever is lower). If they do, follow the recommendations of the instrument manufacturer to correct for this condition. Begin the sample run by flushing the system with the reagent blank solution between each sample. It is recommended to analyse a calibration check solution and the calibration blank solution every 10 samples. Analyze the sample solution and calculate the concentration in $\mu\text{g/ml}$ of the calcium and magnesium in the sample solution.

NOTE — It is recommended that IS 3025(Part 2)/ISO 11885 may be referred and practiced for ensuring precise and reproducible analysis.

C-6 CALCULATION

The mass concentrations for each element are determined with the aid of the instrument software by following steps:

- Relate emission signals from calibration blank and calibration solutions with the signals from reference elements and establish a calibration plot; and
- Determine the mass concentrations of samples with the aid of the emissions and the calibration graphs and calculate the quantity in mg/kg of the constituent elemental impurities in the sample, by multiplying the value by 20 (dilution factor).

Table 2 Recommended Wavelengths, Achievable Limits of Quantification for Different Configuration of Instruments and Important Spectral Interferences
(Clauses [C-2](#) and [C-4](#))

Sl No.	Element	Wavelength (nm)	Approximately Achievable Limits		Interfering Elements
			Radial Viewing (μg)	Axial Viewing (μg)	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Ca	315.887	100	13	Co, Mo
		317.933	26	4	Fe, V
		393.366	0.4	25	V, Zr
		422.673	–	–	V, Mo, Zr
ii)	Mg	279.078	33	19	Fe
		279.553	1	7	Fe
		285.213	4	14	Cr

ANNEX D

(Foreword)

COMMITTEE COMPOSITION

Inorganic Chemicals Sectional Committee, CHD 01

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Central Salt and Marine Chemicals Research Institute, Bhavnagar	DR KANNAN SRINIVASAN (<i>Chairperson</i>)
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