

भारतीय मानक मसौदा
खनिज जिप्सम — विशिष्टि
(आईएस 1290 का तीसरा पुनरीक्षण)

Draft Indian Standard
MINERAL GYPSUM — SPECIFICATION
(Third Revision of IS 1290)

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ICS 71.060

Inorganic Chemicals Sectional Committee, CHD 01

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FOREWORD

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

This Indian Standard was first published in 1960 and covered mineral gypsum for use in ammonium sulphate and cement industries. It was revised in 1965 after including other grades of mineral gypsum, namely, those used for surgical plaster, pottery and reclamation of soil. The Committee responsible for the preparation of this standard decided to revise it again in the light of the changed pattern of consumption and production in the country. In the previous revision, the range for quality of gypsum for fertilizer industry was specified. For cement industry, gypsum of 70 to 75 percent purity was prescribed in place of 80 to 85 percent in view of the fast depleting reserves of good quality gypsum in the country. Additional requirement of whiteness was prescribed for production of white cement. The grade of gypsum for soil reclamation was deleted as a separate Indian Standard was published for it (*see* IS 6046).

In this revision, instrumental test methods for the determination of aluminium calcium, chlorides, magnesium and iron have been added as alternate test methods. Also, Packing and Marking clause has been updated. Further, Reference clause has been incorporated in this revision.

Fertilizer and cement are the two important industries in which gypsum finds abundant use. High purity gypsum is utilized in large quantities in the manufacture of ammonium sulphate fertilizer. Gypsum of less purity in crushed condition is utilized in portland cement manufacture, where it acts as a retarder, controlling the setting time of cement.

Calcined gypsum finds use in the plasters and manufacture of plaster of paris. It is also used in the manufacture of partition blocks, sheets and tiles, plaster and insulating boards, and for stucco and lattice works. Ground gypsum blocks are used as building stones. In pottery, gypsum is used for moulding purposes. Besides, gypsum rock is used as flux in the smelting of nickel ores and in tin plate industry for polishing plates.

Alabaster, a massive variety of gypsum is employed for statuary purpose while the silky fibrous variety known as satin spar is employed for making small ornamental articles. Selenite, a crystalline variety is used to a limited extent for gypsum plate in microscopes. Low grade gypsum finds use in the manufacture of gypsum wall boards.

The quality of mineral gypsum available in India is very inconsistent and the percentage of calcium sulphate varies even in one and the same deposit. The ratio between the high grade gypsum of above 80 percent calcium sulphate content and the rest is generally 1 : 3. Consequently, a huge amount of low grade gypsum for which economical beneficiation arrangements are not available at present, remains untapped causing considerable difficulty to the mining industry. To offset the national wastage involved, it is essential that a particular industry should derive its supply of the necessary quality of gypsum and not of a higher or a lower quality. This standard is intended to guide the trade in using the right quality of gypsum for use in the manufacture of surgical plaster, ammonium sulphate, pottery and cement.

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.

1 SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for mineral gypsum. It also covers selenite, a colourless and transparent variety of gypsum which occurs as distinct monoclinic crystals, especially in clay rocks.

1.2 This standard does not cover marine gypsum obtained from marine brine which is covered under a separate Indian Standard (*see* IS 4256).

2 REFERENCES

2.1 The Indian Standards listed below contain provisions which through reference in this text, constitute provisions of this Indian Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<i>IS No.</i>	<i>Title</i>
1070:1992	Reagent Grade Water — Specification (<i>third revision</i>)
1288: 1982	Methods of test for mineral gypsum (<i>second revision</i>)
1289:1960	Methods for sampling of mineral gypsum
4256: 1967	Hydrated calcium sulphate from marine brine — Specification (<i>first revision</i>)
6046: 1982	Specification for gypsum for agricultural use (<i>first revision</i>)

3 TYPES

3.1 The material shall be of the following four types:

- Type 1* — for surgical plaster industry,
- Type 2* — for ammonium sulphate industry,
- Type 3* — for pottery industry, and
- Type 4* — for cement industry.

4 REQUIREMENTS

4.1 The material shall be the natural mineral consisting essentially of hydrated calcium sulphate and free from added impurities.

4.2 The material shall comply with the requirements prescribed in Table 1, when tested according to the methods prescribed in IS 1288. Reference to the relevant clauses of IS 1288 is given in col 7 of the table.

4.3 Additional Requirements

4.3.1 Acidity and Alkalinity

Type 1 of the material shall also pass the test prescribed in Annex A.

4.3.2 Sieve Analysis

Type 2 of the material shall conform to the sieve analysis as agreed to between the purchaser and the supplier.

4.3.3 For use in white cement, Type 4 of the material shall also be tested for whiteness and iron content. The limits and methods of test for these two characteristics shall be as agreed to between the purchaser and the supplier.

4.3.4 Filtrability

The material of Type 2 when tested by the method prescribed in 15 of IS 1288, shall have filtrability time of 50 s maximum.

Table 1 Requirements for Mineral Gypsum
(Clause 4.2)

Sl. No.	Characteristic	Requirement For				Method of Test (Ref to CI No.)
		Type 1	Type 2	Type 3	Type 4	
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	Free water, percent by mass, <i>Max</i>	1.0	—	1.0	—	CI 4 of IS 1288
ii)	Carbon dioxide (as CO ₂), percent by mass, <i>Max</i>	1.0	—	3.0	—	CI 7 of IS 1288
iii)	Silica and other insoluble matter, percent by mass, <i>Max</i>	0.7	6.0	6.0	—	CI 8 of IS 1288
iv)	Iron and aluminium (as oxides), percent by mass, <i>Max</i>	0.1	1.5	1.0	—	A-4 & CI 9 of IS 1288
v)	Magnesium oxide (as MgO), percent by mass, <i>Max</i>	0.5	1.0	1.5	3.0	A-4 & CI 11 of IS 1288
vi)	Calcium sulphate (as CaSO ₄ .2H ₂ O), percent by mass	96.0, <i>Min</i>	85-90	85.0, <i>Min</i>	70-75	A-4 & CI 12 of IS 1288
vii)	Chlorides (as NaCl), percent by mass, <i>Max</i>	0.01	0.003	0.1	0.5	A-3 & CI 14 of IS 1288

5 PACKING AND MARKING

5.1 Packing

The material shall be supplied in bulk or in packages as agreed to between the purchaser and the supplier.

5.2 Marking

When supplied in packages, each package shall be securely closed and marked indelibly with the following information:

- a) Name and type of the material;
- b) Mass of the material in the package;
- c) Manufacturer's name and/or recognized trade-mark, if any; and
- d) Lot number.

5.2.1 When supplied in bulk, a good sized metallic or cardboard label bearing the above information with suitable paint or ink shall be conspicuously displayed on the carrier and also placed inside.

5.2.2 *BIS Certification Marking*

The product may also be marked with the Standard Mark.

5.2.2.1 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.

6 SAMPLING

6.1 Preparation of Test Samples

Representative samples of the material shall be drawn as prescribed in IS 1289.

6.2 Number of Tests

Tests for the determination of calcium sulphate content and silica and other insoluble matter shall be performed on each of the individual sample.

6.3 Criteria for Conformity

6.3.1 *For Individual Samples*

For each of the characteristics tested on the individual samples, the mean (\bar{X}) and range (R) of test results shall be computed separately (range being defined as the difference between the maximum and the minimum of the test results).

6.3.1.1 The lot shall be declared as conforming to the requirements for the characteristics tested on the individual samples if the conditions given below are satisfied:

- a) For silica and other insoluble matter:

$$X + 0.6 R \leq \text{the maximum value specified against SI No. (iii) of Table 1.}$$

- b) For calcium sulphate:

$$X - 0.6 R \geq \text{the minimum value specified against SI No. (vi) of Table 1.}$$

6.3.2 *For Composite Sample*

For declaring the conformity of the lot to the requirements of the characteristics tested on the composite sample the test result for each characteristic shall satisfy the corresponding requirements specified in Table 1.

6.3.3 The lot shall be considered to be conforming to all the requirements of this specification if it satisfies the conditions in **6.3.1** and **6.3.2**, otherwise not.

7 TEST METHODS

7.1 Test shall be conducted according to the methods prescribed in IS 1288 and Annex A of this standard. References to relevant clauses of IS 1288 and Annex A are given in **4.2** and **4.3**.

7.2 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.

ANNEX A

(*Clause 4.3.1*)

TEST FOR ACIDITY AND ALKALINITY

A-1 REAGENTS

A-1.1 Methyl Orange Indicator Solution – Dissolve 0.1 g of methyl orange in 100 ml of water.

A-1.2 Phenolphthalein Indicator Solution — Dissolve 0.01 g of phenolphthalein in 100 ml of 60 percent rectified spirit.

A-2 PROCEDURE

A-2.1 Shake 5 g of the prepared sample (*see* **5** of IS 1288) with 20 ml of water. Warm the mixture to about 40°C and then filter. Test the clear filtrate with methyl orange and phenolphthalein indicator solutions.

A-2.2 The material shall be considered as having passed the test if the filtrate is neither acidic to methyl orange nor alkaline to phenolphthalein.

A-3 TEST METHOD FOR THE DETERMINATION OF CHLORIDES

A-3.1 OUTLINE OF THE METHOD (METHOD A)

This test method covers the determination of sodium chloride in gypsum and gypsum products by the colorimetric method. The presence of sulfide sulfhydryl, or other silver reactive substances will lead to high results. Such interfering substances are removed by alkaline oxidation with hydrogen peroxide.

A-3.1.1 Apparatus

A-3.1.1.1 Chloride Meter

The instrument shall be quipped to measure the concentration of dissolved chloride in aqueous solutions by the colorimetric method. The instrument shall be capable of measuring chloride concentrations in the range from 10 to 260 mg/l with are peat ability of ± 1 mg/l.

A-3.1.1.2 *Magnetic Stirrer with hot plate*

A-3.1.1.3 *Filter Paper* — Ashless whatman filter paper No. 42.

A-3.1.1.4 *Analytical Balance* — capable of weighing 1 mg at least

A-3.1.2 *Reagents***A-3.1.2.1** *Acid Buffer Solution*

Dissolve 100 ml of 99.5 percent acetic acid (CH_3COOH) and 5.5 ml of concentrated nitric acid (specific gravity 1.42) in approximately 200 ml of water and dilute to 500 ml.

A-3.1.2.2 *Diluted Standard Solution (100 mg Cl/l)* — Dilute 5.00 ml of stock standard solution to 500 ml.

A-3.1.2.3 *Gelatin Solution*

Add 2.5 g of gelatin and 0.5 g of thymol blue to 250 ml of water and dissolve by stirring continuously while bringing to a boil. With the solution just boiling, continue stirring until all the thymol blue is dissolved Add 0.5 g of thymol, cool, and dilute the solution to 500 ml.

NOTE—The gelatin solution holds the precipitated silver chloride (AgCl) in suspension and also indicates the presence of the acid buffer. The solution will keep for 3 months at room temperature or longer if refrigerated. Warm the refrigerated solution to room temperature before use.

A-3.1.2.4 *Stock Standard Solution (10 g Cl/l)*

Dissolve 8.240 g of dried sodium chloride (NaCl) in water and dilute to 500 ml.

A-3.1.3 *Procedure*

Weigh 20.0 g of the well-mixed sample and transfer to a 150 ml beaker. Add 50 ml of water, boil, allow the solid material to settle, and filter off the solution. Add an additional 50 ml of water to the solids, boil, and pour the contents of the beaker into the filter. Wash the residue with 100 ml of hot water, adding the washing to the filtrate. Cool and dilute with water to 250 ml.

Switch on the chloride meter and allow a period of 25 min before use. Set the counter to zero. Place a magnetic stirring bar in the test beaker, add 10 ml of diluted standard solution, 3 ml of acid buffer solution and 5 drops of gelatin solution. Place the test beaker on the platform and lower the electrodes into the solution. Press the “start” button until the pilot light is extinguished. The counter will begin to register after a few seconds. Do not remove the electrodes from the sample until the pilot light comes on. Read the chloride content from the counter .If are adding of 100 ± 1 mg Cl/l is not obtained, refer to the manufacturer’s instruction manual. Reset the counter to zero.

Repeat the procedure, using 10 ml of the sample solution instead of the diluted standard solution. Read the result as milligrams of chlorine per litre. When all tests are completed, lower the electrodes into reagent water.

A-3.1.4 *Calculation*

Calculate the amount of NaCl as a percentage of the sample as received or dried sample as follows:

$\text{NaCl, percent by mass} = 0.00206 \times A$

where

A = chloride meter reading, mg Cl/l.

A-3.2 ION CHROMATOGRAPHY FOR CHLORIDES (METHOD B)

A-3.2.1 Principle

Ion Chromatography is an innovative method for the determination of ions. The technique is used for the analysis of chlorides. The technique separates ions and polar molecules based on their affinity to ion exchanger. When the method is employed for the determination of the anions, the identification should be made by using a matrix covering the ions of interest. In cation exchange chromatography, the stationary phase is functionalized with anions. These anions will attach cations towards it. These surface bound molecules/ionic species can then be removed by using a suitable eluent containing substituted ions to replace them or they can be removed by changing the pH of the column. Similarly, in anion exchange chromatography, the stationary phase is cationic in nature. These cations will then separate the anions.

Conductivity detector is generally used in this method. In case of suppressor ion exchange chromatography, analyte ions are separated on the ion exchange column and these ions together with the eluent move to the matrix suppressor. The eluent conductivity is lowered in the suppressor and the sample ion conductivity is increased leading to the large increase in signal to noise ratio.

A-3.2.2 Equipment

A-3.2.2.1 Anion guard column — a protector of the separator column.

A-3.2.2.2 Anion Separator column — suitable for selective separation of ions under analysis.

A-3.2.2.3 Anion Suppressor device

Anion micromembrane suppressor is used to analyse the data Detector: Conductivity detector.

A-3.2.2.4 Software

Software suitable for control of various operating parameters, receiving inputs and analysis of all data.

Sample loop of 100 μl , 200 μl , 500 μl or 1000 μl be used to determine ionic concentration as per instrument manual and practice.

A-3.2.3 Reagents and Standards

A-3.2.3.1 Glass or polyethylene sample bottles.

A-3.2.3.2 Distilled water or deionized water free from the anions of interest.

A-3.2.3.3 Eluent — 1.7 mM of sodium bicarbonate and 1.8 mM of sodium carbonate solution is used.

For preparation of these solution, 0.2856 g of sodium bicarbonate and 0.3816 g of sodium carbonate is dissolved in 2 l of water.

A-3.2.3.4 *Micromembrane suppressor solution (0.025 N of sulphuric acid)*

Dilute 2.8 ml of conc. Sulphuric acid in 4 l of water

A-3.2.4 *Standard solutions*

A-3.2.4.1 *Chloride* — Dissolve NaCl, 1.6485 g in 1 l of reagent water

A-3.2.5 *Calibration and Standardization*

For each analyte of interest, prepare calibration standards at three concentration levels and a blank by adding measured stock standards and diluting with reagent water. If the concentration of the sample exceeds the calibration range, the sample may be diluted. Using 0.1-1.0 ml injections of each calibration standard, tabulate area responses or peak height against the concentration. Use these results to prepare calibration curve. Record the retention time during the procedure.

A-3.2.6 *Procedure*

Dissolve between 1 to 5 g sample in 25 ml reagent grade water in PTTE/HDPE beaker and use this solution for analysis. Inject a well-mixed sample (0.1-1.0 ml) and flush it through an injection loop using each new sample. Use the loop of same size for the standards and samples. Record the peak in size and area units. An automated constant volume injection system may preferably be used. The width of peak for retention time of ions should be same for sample and standard and deviation of retention force shall not exceed ± 10 percent of RT of calibration. Dilute the sample with the help of reagent water if the response for the peak exceeds the working range of the system for analysis. If required, spike the sample with an appropriate amount of standard and reanalyze in case of absence of distinct resolution. Retention time is inversely proportional to concentration. For clear resolution, the sample can further be diluted. The dilution should be made to an extent till there is no deviation from the method.

A-3.2.7 *Data analysis and Calculations*

Prepare a calibration curve for each analyte by plotting instrument response against concentration. Compare the sample response with the standard curve and compute sample concentration. Multiply the value by appropriate dilution factor.

Report results in mg/l or by suitably modifying into percentage. Only report those values that fall within the range of lowest and highest calibration standards.

A-4 DETERMINATION OF ALUMINIUM, IRON, CALCIUM, MAGNESIUM BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD**A-4.1 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.

A-4.2 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.

Table 2 Recommended Wavelengths, Achievable Limits of Quantification for Different Configuration of Instruments and Important Spectral Interferences

Sl No.	Element	Wavelength (nm)	Approx. Achievable limits		Interfering Elements
			Radial viewing (µg)	Axial viewing (µg)	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Fe	238.204	14	(3)	Co
		259.940	6	2	Co
		271.441	-	-	-
ii)	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
iii)	Al	167.079	1	2	Fe, Pb
		308.215	100	17	Fe, Mn, OH, V
		396.152	10	6	Cu, Fe, Mo, Zr
iv)	Mg	279.078	33	19	Fe
		279.553	1	7	Fe
		285.213	4	14	Cr

A-4.3 Reagents and Solutions

A-4.3.1. Nitric acid (65 percent) Suprapure

A-4.3.2 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, 100 or 1000 µg/ml of Iron, calcium, magnesium, and aluminium in 2-5 percent nitric acid. It is preferable to prepare single stock solution of multi elemental standards for analysis.

A-4.3.3 Standard solution

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

A- 4.3.4 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

A-4.3.5 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, polytetrafluorethylene (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.

A-4.4 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

A-4.5 Procedure

A-4.5.1 Calibration

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions (A-4.3.5). 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.

A-4.5.2 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 lead (and/or Iron, calcium, magnesium, manganese, arsenic, molybdenum, aluminium and mercury) in the sample solution.

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A-4.6 Calculation

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

- i) Relate emission signals from calibration blank and calibration solutions with the signals from reference elements and establish a calibration plot.
- ii) 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).