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कृषि उपयोग के लिए जिप्सम — विशिष्टि  
(दूसरा पुनरीक्षण)

**Gypsum for Agricultural Use  
— Specification**  
( *Second Revision* )

ICS 65.080

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## FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Soil Quality and Fertilizers Sectional Committee had been approved by the Food and Agriculture Division Council.

The main soil amendment for alkali soils available in the country is the mineral gypsum. This agricultural grade gypsum is mined at several places in the country but the bulk of the quantity occurs in western Rajasthan.

This standard was originally published in 1971 and first revised in 1982 in the light of experience gained in the use of mineral gypsum as soil amendment. In the first revision of the standard, the minimum limit for calcium sulphate dihydrate content was increased from 50 percent to 70 percent to facilitate procurement of quality gypsum for use as soil amendment.

In this second revision, the major changes include:

- a) Three grades of mineral gypsum have been prescribed based on calcium sulphate content to take into account the variation in the intrinsic content of calcium sulphate in the soil of different agro climatic zones of the country; and
- b) Amendment No. 1 and 2 to the previous revision have been incorporated.

Committee responsible for the formulation of this standard is listed in Annex C.

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***GYPSUM FOR AGRICULTURAL USE — SPECIFICATION***( Second Revision )***1 SCOPE**

This standard prescribes the requirements, packing and marking, methods of sampling and testing of gypsum for use as an amendment for alkali soils. It covers only mineral gypsum.

**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 standard are encouraged to investigate the possibility of applying the most recent editions of these standards.

**3 REQUIREMENTS****3.1 Fineness**

All the material shall pass through a 2 mm sieve and at least 50 percent of it should pass through a 0.25 mm (60 mesh) sieve [see IS 460 (Part 1)], when tested by the method prescribed in 3 of IS 1288.

**3.2** The material shall also confirm to requirements given in Table 1.

**4 PACKING AND MARKING****4.1 Packing**

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

**4.2 Marking**

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

- a) Manufacturer's name and address with recognized trade-mark;
- b) Name of the material (that is, gypsum for agriculture use);
- c) Net quantity of the material in the package;
- d) Minimum calcium sulphate dihydrate content;
- e) Particle size; and
- f) Lot number and date of manufacture.

**Table 1 Requirements for Gypsum**  
(Clause 3.2)

Sl No.	Characteristics	Requirement			Method of Test, Ref To
		Grade 1	Grade 2	Grade 3	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Calcium sulphate dihydrate, percent by mass, <i>Min</i>	50	60	70	Annex B
ii)	Sodium content, percent by mass (as Na), <i>Max</i>	0.75	0.75	0.75	Annex C

**4.2.1** When supplied in bulk, a metallic or card board label of appropriate size, bearing the information required to be given under 4.2 with suitable paint or ink shall be conspicuously displayed on the bulk carrier and also placed inside the consignment.

**4.2.2 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**

Representative test samples of the material shall be drawn as given in 5 of IS 1289.

**6 TESTS**

**6.1** Tests for all the characteristics given in 3 shall be conducted on the composite samples.

**6.2 Criteria for Conformity**

**6.2.1** A lot shall be considered in conformity to the specification if the composite sample meets all the requirements given in 3.

**ANNEX A**  
**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 1113 : 1965	Specification for ammonium chloride, technical and pure
IS 265 : 2021	Hydrochloric acid — Specification ( <i>fifth revision</i> )	IS 1288 : 1982	Methods of test for mineral gypsum ( <i>second revision</i> )
IS 266 : 1993	Sulphuric acid — Specification ( <i>third revision</i> )	IS 1289 : 1960	Methods for sampling of mineral gypsum
IS 460 (Part 1) : 2022	Test sieves — Specification: Part 1 Wire cloth test sieves ( <i>fourth revision</i> )		

**ANNEX B**

[Table 1, Sl No. (i)]

**DETERMINATION OF CALCIUM SULPHATE CONTENT**

**B-1 GENERAL**

For determining calcium sulphate content, determine calcium content and sulphate content separately.

**B-2 CALCIUM**

**B-2.1 General**

Calcium is precipitated as calcium oxalate which is titrated against potassium permanganate.

**B-2.2 Reagents**

**B-2.2.1 Ammonium Acetate-Acetic Acid Buffer**

Dissolve 7.708 g ammonium acetate in 100 ml of distilled water and mix with 100 ml of 1 N acetic acid to get a buffer of pH 4.5.

**B-2.2.2 Ammonium Chloride** — see IS 1113.

**B-2.2.3 Ammonium Hydroxide** — approximately 4 N.

**B-2.2.4 Ammonium Oxalate** — Pure.

**B-2.2.5 Bromophenol Blue 0.04 Indicator** — percent solution in distilled water.

**B-2.2.6 Concentrated Hydrochloric Acid** — see IS 265.

**B-2.2.7 Concentrated Nitric Acid** — see IS 264.

**B-2.2.8 Dilute Ammonium Hydroxide** — 1 N.

**B-2.2.9 Dilute Hydrochloric Acid** — approximately 4 N.

**B-2.2.10 Dilute Potassium Permanganet Solution** — 0.1 Percent.

**B-2.2.11 Dilute Sulphuric Acid** — approximately 4 N.

**B-2.2.12 Methyl Red Indicator**

Dissolve 0.15 g of methyl red in 500 ml of water.

**B-2.2.13 Standard Potassium Permagnet Solution** — 0.05 N.

**B-2.3 Procedure**

**B-2.3.1 Preparation of Sample**

Crush the material so as to pass through a 1 mm IS sieve. Weigh 5 g of the sieved material and place it in a thin layer in a petridish. Dry in an oven maintained at  $(45 \pm 1)^\circ\text{C}$  for two hours and then cool in a desiccator. This shall be the prepared sample.

**B-2.3.2** Weigh accurately 0.5 g of the prepared sample in a porcelain dish. Add 25 ml of dilute hydrochloric acid and evaporate on a low flame to almost dryness. Cool and add enough of concentrated hydrochloric acid to wet the residue thoroughly. Add 10 ml of water and boil. Filter and wash the residue thoroughly with water, collecting the filtrate and washing in a beaker. Discard the residue. Add to the filtrate a few drops of concentrated nitric acid and boil to ensure oxidation

of iron, if present. Add 2 g of ammonium chloride previously dissolved in water and then make alkaline with ammonium hydroxide (*see* Note). Boil for a few minutes until the precipitate, if produced, coagulates. Filter and then wash the precipitate three or four times with water, collecting the filtrate and washings in a beaker. Discard the precipitate.

NOTE — In case gypsum contains phosphate ions and for their removal the solution after addition of ammonium chloride should be made alkaline with dilute ammonium hydroxide using bromophenol blue indicator solution. Then add dilute hydrochloric acid dropwise to make the solution slightly acidic (yellow colour of the indicator). Dilute the solution to 200 ml and boil it. Add 20 ml of ammonium acetate-acetic acid buffer solution of pH 4.5 and keep the solution hot for 15 minutes. Filter the precipitate, if produced, through a filter paper and wash it twice, collecting the filtrate and washings in a beaker. Discard the precipitate and proceed as directed in B-2.3.3.

**B-2.3.3** To the filtrate and washings obtained in B-2.3.2, add 2 to 3 drops of methyl red indicator and then dilute hydrochloric acid in slight excess. Make up to 500 ml and then take 125 ml of the solution in a beaker. Heat nearly to boiling and add 2 g of ammonium oxalate previously dissolved in water. To the hot solution, constantly stirred, add ammonium hydroxide drop wise until the mixture is distinctly alkaline. Allow the precipitate to stand for 2 hours. Decant the supernatant through the same filter paper, then wash the precipitate by decantation three or four times. Transfer the precipitate to the filter paper and wash the precipitate and the filter paper first with dilute ammonium hydroxide and then with cold water until 1 ml of the washings does not decolorize a drop of potassium permanganate solution in presence of 1 ml of dilute sulphuric acid. Throw away the filtrate and washings, puncture the filter paper and transfer the precipitate through the puncture into a beaker using a jet of water. Wash the filter paper five times with 10 ml of hot dilute sulphuric acid, collecting the washings in a conical flask and add 10 ml of dilute sulphuric acid and warm to 60 °C. Titrate with standard potassium permanganate solution. As the end point reaches, put the filter paper in the conical flask and continue the titration till end point is restored. Carry out a blank titration on the reagents used.

#### B-2.4 Calculation

$$\text{Calcium content, percent by mass} = \frac{8.0 \times (V - v) N}{M}$$

where

$V$  = volume, in ml, of standard potassium permanganate solution used;

$v$  = volume, in ml, of standard potassium permanganate solution used in blank titration;

$N$  = normality of standard potassium permanganate solution; and

$M$  = mass in g of the prepared sample taken for the test in B-2.3.2.

### B-3 SULPHATE

#### B-3.1 General

Sulphate content is calculated from the mass of barium sulphate precipitate obtained from the sample.

#### B-3.2 Reagents

**B-3.2.1 Barium Chloride Solution** — Dissolve 12 g of barium chloride dihydrate in 100 ml of water.

**B-3.2.2 Concentrated Nitric Acid** — *see* IS 264.

**B-3.2.3 Concentrated Sulphuric Acid** — *see* IS 266.

**B-3.2.4 Dilute Hydrochloric Acid** — approximately 3 N.

#### B-3.3 Procedure

Dissolve 0.2 g of the prepared sample, accurately weighed, in 50 ml of dilute hydrochloric acid. Boil the solution and add 100 ml of boiling water. Continue boiling for 5 minutes. Filter immediately and wash thoroughly with hot water, collecting the filtrate and washings in a 500 ml beaker. Boil the solution in the beaker and, while boiling add 20 ml of hot barium chloride solution in a slow stream with constant stirring. Digest the precipitate in the beaker in a hot water bath for half an hour and then allow settle for 4 h. Filter by decantation through a filter paper (Whatman No. 42 or equivalent) and then wash the precipitate with hot water till washings are free from chlorides. Dry the residue. Ignite the filter paper over low flame in a crucible and when the carbon is burnt away, heat strongly till a white ash is obtained. Add one or two drops of concentrated nitric acid, heat gently to drive off the acid, cool and add one or two drops of concentrated sulphuric acid, heat gently at first to drive off the acid and then strongly (to 700 °C). Cool in a desiccator and weigh.

#### B-3.4 Calculation

a) Sulphate content, percent by mass =  $\frac{41.2 \times m}{M}$

b) Calcium sulphate dihydrate (CaSO<sub>4</sub>·2H<sub>2</sub>O),

$$\text{percent by mass} = \frac{73.85 \times m}{M}$$

where

$m$  = mass in g of the precipitate;

$M$  = mass in g of the prepared sample taken for the test.

## ANNEX C

[Table 1, Sl No. (ii)]

## DETERMINATION OF SODIUM CONTENT

**C-1 GENERAL**

Two methods, namely, Method A and Method B, are given. In case of dispute, Method A shall be followed.

**C-2 METHOD A (GRAVIMETRIC METHOD)**

**B-2.1** After removing sulphates by adding barium chloride and removing excess of barium by ammonium carbonate, potassium is precipitated as perchlorate and in the filtrate sodium is precipitated as sodium magnesium uranyl acetate and the precipitate weighed.

**C-2.2 Reagents**

**C-2.2.1** *Ammonium Carbonate Solution* — 10 Percent.

**C-2.2.2** *Ammonium Hydroxide* — 2 N.

**C-2.2.3** *Barium Chloride Solution* — 12 Percent

**C-2.2.4** *Concentrated Hydrochloric Acid* — see IS 265.

**C-2.2.5** *Ethanol* — 96 Percent.

**C-2.2.6** *Magnesium Uranyl Acetate Solution*

**C-2.2.6.1** Dissolve 90 g of crystallized uranyl acetate in 60 ml of glacial acetic acid and sufficient water by stirring and warming it to 70 °C, and dilute the solution to 1 000 ml.

**C-2.2.6.2** Dissolve 600 g of crystallized magnesium acetate in 60 ml of glacial acetic acid and sufficient water by stirring and warming to 70 °C, and dilute the solution to 1 000 ml.

**C-2.2.6.3** Mix the two solutions prepared in **C- 2.2.6.1** and **C-2.2.6.2**. Allow to stand for several hours. Filter off any residue. The final solution should be kept at 20 °C in flasks made of glass with very low sodium content. The solution should also be used at 20 °C.

**C-2.2.7** *Perchloric Acid*

**C-2.2.8** *Washing Alcohol A*

Prepared by mixing ethanol with 0.2 percent of perchloric acid and saturated with potassium perchlorate.

**C-2.2.9** *Washing Alcohol B*

Prepared by saturating ethanol with sodium magnesium uranyl acetate precipitate.

**C-2.3 Procedure**

**C-2.3.1** Weigh accurately 5 g of the prepared sample (*see B-2.3.1*), add a few milliliters of concentrated hydrochloric acid and 100 ml of water and heat to boiling in a beaker. To the boiling solution, add slowly in small quantities an excess of barium chloride solution. Heat for some time and then cool. Transfer to a 200 ml volumetric flask and dilute to the mark with water. Stir the solution thoroughly and filter. Reject the first few milliliters of filtrate. Take exactly 20 ml of the filtrate and add a slight excess of ammonium carbonate solution. Filter off the precipitated carbonates and wash the precipitate with water, the washings being added to the filtrate. Evaporate the filtrate and washings to dryness in a porcelain dish of 10 cm diameter and calcine gently. Add to the residue a small quantity of water and 6 ml of perchloric acid and evaporate almost to dryness in a water-bath.

**C-2.3.2** Cool the residue, add a few milliliters of washing alcohol A and crush the moist mass to a fine state by using a glass pestle, Decant off the liquid. Repeat the crushing of the residue and decantation with further additions of washing alcohol A, collecting all the decanted liquid. Transfer the precipitate to a small filter and wash thoroughly with ethanol, adding these washings also to the decanted liquid. Neutralize the filtrate and washings with ammonium hydroxide and heat to dryness. Transfer the residue to a beaker with about 5 ml of water. The solutions should not contain more than 25 mg of sodium (as Na). Add magnesium uranyl acetate solution rapidly (100 ml if the amount of sodium is less than 10 mg and 10 ml/mg for larger amounts), maintaining the temperature at 20 °C. Stir the precipitate and allow to stand for an hour at 20 °C. Decant off the clear solution through a sintered glass crucible which has been previously washed with ethanol, dried at (120 ± 5) °C and weighed. Add a small quantity of washing alcohol B, and repeat the decantation twice. Finally, wash the precipitate with washing alcohol B on the filter. Dry the crucible with the precipitate for half an hour at 105 °C to 110 °C, cool in a desiccators and weigh.

### C-2.4 Calculation

#### C-2.4.1 Sodium content, percent by mass

$$= \frac{1.528 m}{M}$$

where

$m$  = mass in g of the precipitate, and

$M$  = mass in g of the prepared sample taken for the test.

## C-3 METHOD B (FLAME PHOTOMETER METHOD)

### C-3.1 Apparatus

#### C-3.1.1 Flame Photometer

#### C-3.1.2 Glassware

Rinse all glassware with nitric acid (1 : 15) followed by several portions of deionized water to avoid contamination errors.

### C-3.2 Reagents

In order to minimize sodium pickup, all solutions shall preferably be stored in plastic bottles. The use of small containers reduces the amount of sodium which may be picked up from the bottle walls when the solution is poured. Each container shall be thoroughly shaken to wash away accumulated salts from the walls before the solution is poured.

#### C-3.2.1 Deionized Distilled Water

Prepare by passing distilled water through a mixed bed of ion-exchange resins. Use deionized water for the preparation of all reagents, calibration standards and as dilution water.

**C-3.2.2** Weigh 2.542 2 g of Sodium Chloride AR previously dried at 110 °C for one and half hours and cooled in a desiccator in a 1 000 ml standard flask. Dissolve in small volume deionized distilled water and then make up to the volume by the same quality of water. This solution gives 1 000 µg/ml Na which could be stored for about two months.

#### C-3.2.3 Intermediate Standard Sodium Solution

Transfer 25 ml of stock solution to 250 ml standard flask and make up to a volume with deionized distilled water to give 100 µg/ml Na.

#### C-3.2.4 Working Standard Sodium Solution

Take 1 ml, 5 ml, 10 ml, 15 ml and 20 ml of

intermediate solution to 5 different 100 ml standard flasks and make up the volume by deionized distilled water to give 1 µg/ml, 5 µg/ml, 10 µg/ml, 15 µg/ml and 20 µg/ml Na respectively.

### C-3.3 Procedure

#### C-3.3.1 Precautions

Install the flame photometer in an area away from direct sunlight or the constant light emitted by an overhead fixture, and free of draughts, dust and tobacco smoke. Guard against contamination arising from corks, filter paper, perspiration, soap, cleaning mixtures, and inadequately rinsed apparatus.

#### C-3.3.2 Instrument Operation

Because the differences between the makes and models of satisfactory flame photometers render impossible the formulation of detailed instructions applicable to every instrument, follow the manufacturer's recommendation for the selection of the proper photocell, wave length, adjustment of the slit width and sensitivity, the appropriate fuel, air or oxygen pressures, the steps for warm up, correcting for flame background, rinsing of the burner, ignition of sample, and measurement of the emission intensity.

**C-3.3.3** Use deionized distilled water as a blank to set zero and 10 µg/ml sodium standard for maximum deflection, for example 100. Once the instrument is checked for 0 and 100, use the working standards to prepare a calibration curve. Starting with the highest calibration standard and working towards the most dilute, measure the emission using sodium filter. For test with the sample, use the solution containing sodium (if present) prepared in C-2.3.2 and dilute it sufficient so as to obtain a sodium concentration within the range of the calibration standards. Measure the emission with both the calibration standards and the samples repeatedly sufficient number of times to secure a reliable mean reading for each solution. Draw a calibration graph from the sodium standards. Determine the sodium concentration of the sample by means of the calibration graph.

**C-3.4** Calculate the sodium content present in the original sample by multiplying the value read from the calibration graph by the appropriate dilution factor.

**ANNEX D**  
(Foreword)

**COMMITTEE COMPOSITION**

Soil Quality and Fertilizers Sectional Committee, FAD 07

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### Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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