BUREAU OF INDIAN STANDARDS

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भारतीय मानक *मसौदा*

उपोत्पाद जिप्सम, कृषि ग्रेड – विशिष्टता

(*आइ एस 10170 का पहला पुनरीक्षण*)

***Draft** Indian Standard*
BYPRODUCT GYPSUM, AGRICULTURAL GRADE - SPECIFICATION

(*first revision of IS 10170*)

ICS 65.080

Soil Quality and Fertilizers Sectional Committee, FAD 07	Last date of comments: **15 November 2024**

FOREWORD

(*Formal clauses would be added later*)

Byproduct gypsum is produced in the country in phosphoric acid plants following wet process technology. This byproduct gypsum like mineral gypsum is also a major soil amendment for reclamation of alkali soils. A separate Indian Standard on mineral gypsum for agriculture use, IS 6046 : 2023 has also been published.

This standard was originally published in 1982. In this revision, the following major changes have been made:

a) The requirement of fineness has been updated to align it with *Fertilizer (Control) Order, 1985*; and

b) The requirements of sulphur and heavy metals (Lead, Cadmium, Chromium and Arsenic) along with their corresponding test methods have been incorporated in line with *Fertilizer (Control) Order*, 1985.

In the preparation of this standard, due consideration has been given to the *Fertilizer* (*Control*) *Order* (*FCO*), 1985 and *Legal Metrology* (*Packaged Commodities*) *Rules*, 2011. However, this standard is subject to restrictions imposed under these, wherever applicable.

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

This standard prescribes the requirements and methods of sampling and test for byproduct gypsum in powdered and granular forms used as an amendment for alkali soils.

2 REFERENCES

The standards listed below 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 the standards listed below:

IS No.	Title
IS 265 : 2021	Hydrochloric acid — Specification (*fifth revision*)
IS 266 : 2024	Sulphuric acid — Specification (*fourth revision*)
IS 1288 : 1982	Methods of test for mineral gypsum (*second revision*)
IS 6046 : 2023	Gypsum for agricultural use — Specification (*second revision*)
IS 6092 (Part 5) : 1985	Methods of sampling and test for fertilizers: Part 5 Determination of secondary elements and micronutrients (*first revision*)
IS 6092 (Part 6) : 1985	Methods of sampling and test for fertilizers: Part 6 Determination of moisture and impurities (*first revision*)
IS 1289 : 1960	Methods for sampling of mineral gypsum

3 REQUIREMENTS

3.1 Fineness

3.1.1 For the powdered form, all the material shall pass through 2 mm sieve and 50 percent of it should pass through 0.25 mm (60 mesh) sieve when tested by the method prescribed in IS 1288.

3.1.2 For granular form, not less than 90 percent of the material shall pass through 4 mm IS sieve and shall be retained on 1 mm IS sieve. Not more than 5 percent shall pass through 1 mm IS sieve when tested by the method prescribed in IS 1288.

3.2 The material shall also conform to the requirements given in Table 1.

Table 1 Requirements for Byproduct Gypsum, Agricultural Grade
(*Clause* 3.2)

Sl. No.	Characteristics	Requirements	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Calcium sulphate dihydrate content, percent by mass, on dry basis, *Min*	70	IS 6046
ii)	Sodium content as (Na), percent by mass, on dry basis, *Max*	0.75	IS 6046
iii)	Fluorine (as F), percent by mass, *Max*	1	Annex A

iv)	Moisture content, percent by mass, *Max*	15	Annex B
v)	Sulphur (as S), percent by mass, on dry basis, *Min*	13	IS 6092 (Part 5)
vi)	Lead (as Pb), mg/kg, *Max*	100	IS 6092 (Part 5)
vii)	Cadmium (as Cd), mg/kg, *Max*	5	Annex C
viii)	Chromium (as Cr), mg/kg, *Max*	50	IS 6092 (Part 6)
ix)	Nickel (as Ni), mg/kg, *Max*	50	IS 6092 (Part 6)
x)	Arsenic (as As_2O_3), mg/kg, *Max*	10	Annex D

4 PACKING

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

5 MARKING

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

a) Name of the material;
b) Quantity of the material in the package;
c) Minimum calcium sulphate dihydrate content;
d) Particle size;
e) Moisture content;
f) Manufacturer's name and recognized trade-mark;
g) Lot number; and
h) Any other requirements as specified under the *Fertilizer* (*Control*) *Order*, 1985 and the *Legal Metrology* (*Packaged Commodities*) *Rules*, 2011.

5.2 When supplied in bulk, a metallic or card board label of appropriate size, bearing the information required to be given under **5.1** with suitable paint or ink shall conspicuously be displayed on the bulk carrier and also placed inside the consignment.

5.3 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.

6 SAMPLING

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

6.2 Number of Tests

6.2.1 Calcium sulphate dihydrate shall be tested on each of the individual samples.

6.2.2 Tests for remaining characteristics given in **3.1.2** and Table 1 of the specification shall be conducted on the composite sample.

6.3 Criteria for Conformity - The lot shall be declared as conforming to the requirements of the specification if **6.3.1** and **6.3.2** are satisfied.

6.3.1 The expression 'X - 0.6 R' is greater than or equal to the minimum limit prescribed in Table 1 of the specification for calcium sulphate dihydrate, where:

$$\text{Mean } (X) = \frac{Sum\ of\ the\ test\ result}{Number\ of\ the\ result}$$

Range (R) = Difference in the maximum and minimum of the test results.

6.3.2 All the test results on the composite sample meet the relevant requirements given in **3.1.2** and Table 1 of the specification.

ANNEX A
[*Table* 1, *Sl No.* (iii)]
DETERMINATION OF FLUORINE CONTENT

A-1 GENERAL

A-1.1 The determination of fluorine in byproduct involves decomposition of insoluble fluorine compounds, distillation with concentrated sulphuric acid and separation of fluorine from the distillate by perchloric acid. The fluorine in the distillate is determined by spectrophotometric method using Zirconium-Eriochrome Cyanin-R Lake.

A-2 REAGENTS

A-2.1 Sulphuric Acid – *see* IS 266.

A-2.2 Sodium Hydroxide – 10 percent and 50 percent solution prepared in double distilled water.

A-2.3 Perchloric Acid – 70 percent.

A-2.4 Silver Perchlorate – 17.5 percent solution prepared in double distilled water.

A-2.5 *p*-Nitrophenol Indicator – 0.5 percent solution prepared in double distilled water.

A-2.6 Erlochrome Cyanin-R

Dissolve 1.80 g of Eriochrome Cyanin-R in double distilled water and dilute to one litre.

A-2.7 Zirconyl Chloride Octahydrate

Dissolve 0.265 g of zirconyl chloride in 50 ml double distilled water, add 700 ml concentrated hydrochloric acid and dilute to one litre with double distilled water.

A-2.8 Concentrated Hydrochloric Acid – *see* IS 265.

A-2.9 Reference Solution

All of Eriochrome Cyanin-R (*see* **A-2.6**) is added to 50 ml volumetric flask, 5 ml of solution prepared by diluting 3 ml of concentrated hydrochloric acid (*see* **A-2.8**) to 5 ml with double distilled water is added to the volumetric flask and the volume is made to 50 ml with double distilled water. The solution is used for setting the reference point (100 percent transmittance) of the spectrophotometer.

A-2.10 Standard Fluoride Solation

Dissolve 2.211 g of sodium fluoride in double distilled water and make the volume to one litre in a volumetric flask. Pipette out 10 ml of this solution into one litre volumetric flask and make up the volume with double distilled water. This solution contains 0.01 mg of fluorine per millilitre.

A-3 APPARATUS

A-3.1 Spectrophotometer

A-3.2 Distillation Apparatus – with three necked distillation flask of 500 ml capacity.

A-3.3 Thermometer – 0 °C to 200 °C range,

A-3.4 Steam Generator – 2 litre capacity.

A-3.5 Steam Condensation Trap – 60 ml capacity.

A-3.6 Electric Heating Mantle – provided with thermostat and to accommodate 500 ml distillation flask.

A-4 PREPARATION OF SAMPLE

10 g of air dried byproduct gypsum sample is pulverized in mortar and pestle until the entire sample passes through 200 mesh sieve. The ground sample is oven dried at 100 °C to a constant mass.

A-5 PROCEDURE

A-5.1 500 mg of the sample (*see* **A-4**) is transferred to the three-necked distillation flask. 50 ml concentrated sulphuric acid and 25 ml double distilled water are added. The distillation flask is placed in an electric heating mantle with thermostat. A thermometer (0 °C to 200 °C) is installed in one of the necks of the distillation flask. The lower 2.5 cm tip of the thermometer is immersed in the concentrated sulphuric acid. The steam distillation head is attached with a 500 ml calibrated beaker placed under the condenser to collect the distillate. The heat is applied slowly to the distillation flask through the heating mantle. When the temperature reaches 160 °C allowed to enter the flask. The temperature is maintained at (165 ± 2) °C throughout the distillation. When approximately 400 ml distillate is collected then 10 ml of 10 percent sodium hydroxide is added to it and evaporated to near dryness on a hot plate. After evaporation of the distillate, 25 ml of double distilled water is added to dissolve the salt residues.

A-5.2 The distillate obtained in operation (*see* **A-5.1**) is transferred quantitatively from the beaker to a three-necked distillation flask with 50 ml of 70 percent perchloric acid and 25 ml double distilled water. One millilitre of 17.5 percent silver perchlorate solution is added to the flask in order to precipitate the chlorides. The distillation flask is connected to steam distillation head and a 500 ml beaker is placed below the condenser to collect the distillation. One drop of 50 percent sodium hydroxide and two drops of *p*-nitrophenol indicator are added in the beaker to make the distillate alkaline. Heat is applied to the distillation flask through an electric heating mantle to raise the temperature to 132 °C. Steam is introduced at this stage and the temperature is raised to 135 °C. The temperature is maintained at (135 ± 2) °C till approximately 400 ml distillate is collected.

A-5.3 The distillate obtained in operation (*see* **A-5.2**) is quantitatively transferred to a volumetric flask and the volume is made to 500 ml with double distilled water,

A-5.4 Take 5 ml portion of the distillate (*see* **A-5.3**) in 50 ml volumetric flask and add 5 ml double distilled water and mix well. Add one drop of p-nitrophenol and then add 4 N hydrochloric acid dropwise till the yellow colour disappears. Add 5 ml of Eriochrome Cyanin-R (*see* **A-2.6**) and 5 ml of Zirconyl Chloride Octahydrate (*see* A-**2.7**) and mix well. Make up the volume to the mark with double distilled water. The colour reaction is immediate and stable, and readings can be

recorded immediately or at any other desired time without significant change in transmittance, provided constant temperature is maintained.

A-5.5 Set the spectrophotometer at 527.5 mm (range 525 mm to 530 mm) for 100 percent transmittance using the reference solution (*see* **A-2.9**) and record the absorbance/transmittance of the sample solution.

A-5.6 Prepare a standard curve for fluorine using standard fluorine solution (*see* **A-2.10**). Standard fluorine solution may further be diluted with double distilled water and aliquots be selected in the range of 0.00 ppm to 1.40 ppm in terms of fluorine for the preparation of standard curve.

NOTE − A new curve should be prepared for each fresh batch of reagents specified in **A-2.6** and **A-2.7**

A-5.7 Calculation

The fluorine value of the sample aliquot is read from the standard curve (*see* **A-5.6**) and is expressed in percentage taking into account the total dilution factor.

ANNEX B
[*Table* 1, *Sl No.* (iv)]
DETERMINATION OF FREE MOISTURE CONTENT

B-1 PREPARATION OF SAMPLE

Reduce gross sample to quantity sufficient for analysis or grind approximately 225 g sample without previous sieving. Grind to pass sieve with one mm circular opening. Grind as rapidly as possible to avoid loss or gain of moisture, Mix thoroughly and store in tightly stoppered bottles.

B-2 PROCEDURE

Weigh 2 g of prepared sample (*see* **B-1**) into a tared glass weighing dish. Dry sample for 2 h ± 10 min at (50 ± 1.5) °C under vacuum of 48 cm to 53 cm. Maintain vacuum by passing desiccated air through chamber. Cool in desiccator and reweigh.

B-3 CALCULATION

$$\text{Free moisture content percent by mass} = \frac{100 \times (A - B)}{A}$$

where

A = mass, in g, of the sample taken for test; and

B = mass, in g, of the material after drying,

NOTE − Absolute pressure of 23 cm to 28 cm temperature control within specified limits throughout oven chamber is essential. In case facility for required vacuum is not, available, dry the sample for 2 h ± 10 min at 70 °C in an oven.

ANNEX C
[*Table* 1, *Sl No.* (vii)]
DETERMINATION OF CADMIUM

C-1 REAGENTS

C-1.1 Standard Cadmium Solution

Weigh out 1 g of pure cadmium metal and transfer it to a 250 ml beaker. Add 50 ml of water and 10 ml of concentrated nitric acid to dissolve the metal completely.

Transfer the cadmium solution to a one litre flask with necessary washing. Make up the volume up to the mark. Shake well. This is a 1000 ppm solution of cadmium, (hereinafter called standard *A*). Dilute 1 ml of standard *A* to 100 ml in a volumetric flask. This is a 10 ppm solution of cadmium, (hereinafter called standard *B*).

C-1.2 Glass distilled water of *p*H (2.5 + 0.5)

Dilute 1 ml of 10 percent sulphuric acid to one litre with glass distilled water and adjust the *p*H to (2.5 + 0.5) with a *p*H meter using sulphuric acid or sodium hydroxide solution. The water so obtained is called acidified water.

C-2 PREPARATION OF WORKING STANDARDS

Pipette out the following volume of standard B in 100 ml of numbered volumetric flask and make up the volume with acidified water. Stopper the flask and shake them well. The same acidified water should be used for the preparation of the sample solution. Fresh standards should be prepared and used every time.

Flask No.	*Volume of standard* (*B*) *taken in ml*	*Concentration of cadmium after making volume to 100 ml* (*in ppm*)
1	0	0.0
2	2.0	0.2
3	4.0	0.4
4	8.0	0.8
5	12.0	1.2
6	16.0	1.6
7	20.0	2.0

C-3 PROCEDURE

C-3.1 Preparation of Sample Solution

Weigh 2 g of sample and transfer it to a 100 ml volumetric flask giving repeated washings with acidified water. Dissolve the material by shaking well, make up the volume and mix thoroughly.

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Filter a portion if necessary. For higher concentration of cadmium adjust the weight and dilution such that the absorbance of final flaming solution is not more than a 2 ppm solution of cadmium.

Aspirate the standards as well as the sample solution in an atomic absorption spectrophotometer at a wave length of 228.8 nm using air acetylene flame and note the corresponding absorbance value for each solution.

C-4 CALCULATION

Draw a graph using concentration (ppm) as the X-axis and absorbance as the Y-axis. Determine the concentration of cadmium in ppm in the sample solution from the graph.

$$\text{Cadmium (as Cd), concentration in ppm} = \frac{C \times F}{M}$$

where

C = concentration, in ppm, of final sample solution
M = mass, in g, of the sample; and
F = the dilution factor.

ANNEX D
[*Table* 1, *Sl No.* (x)]
DETERMINATION OF ARSENIC

D-1 PRINCIPLE

Arsenic V is reduced by KI to Arsenic III and the iodine thus released is reduced in turn by $SnCl_2$. Hydrogen is liberated by the reduction of zinc with HCl in a nascent state. It also converts Arsenic V to Arsenic III. Acidity of the solution plays a key role in this case. The arsenic produced is swept out of the solution as arsine by brisk evolution of hydrogen and it passes through a plug impregnated with lead acetate, which removes any hydrogen sulphide that may be liberated from the solution. H_2S discolours the $HgCl_2$ paper. As the concentration of liberated arsenic as arsine increases, the yellow spot of ($HgCl_2$) turns to brown spot of ($HgCl_3$) and finally to black Hg_3As_2 with further increase in concentration of arsine.

D-2 REAGENT

D-2.1 Concentrated Hydrochloric Acid – A.R. grade

D-2.2 0.5 M HCl – Mix 20 ml concentrated HCl with 420 ml water.

D-2.3 Potassium Iodide Solution – Dissolve 10 g KI in 400 ml water (2.5%).

D-2.4 10 percent Stannous Chloride Solution

Dissolve 25 g $SnCl_2.5H_2O$ in 250 ml concentrated HCl

D-2.5 0.75 percent Stannous Chloride Solution

Mix 75 ml of the 10 percent stannous chloride solution with 375 ml of concentrated hydrochloric acid and 550 ml of water. Do not keep this solution for a period of more than two days.

D-2.6 Ethyl Alcohol (Absolute) – 95 percent.

D-2.7 Mercuric Chloride Solution – Dissolve 25 g $HgCl_2$ in 100 ml of ethyl alcohol.

D-2.8 Filter paper (Whatman No. 40) – 9 cm diameter or comparable grade.

D-2.9 Mercuric chloride paper

Allow 15 Filter paper to soak for 1 h in alcoholic $HgCl_2$ solution. Leave it in the dark to dry, then cut into 1 cm square avoiding the edges of each circle. Store in an air tight and light proof container. Prepare freshly each week.

D-2.10 Lead Acetate Solution – Dissolve 15 g lead acetate trihydrate in 100 ml glacial acetic acid.

D-2.11 Glass Wool – Saturate glass wool with above lead acetate solution and allow it to dry. Pack loose plug of this into the tube of each Gutzeit apparatus (*see* Fig. 1). Replenish when this is discoloured along half of the length of the tube.

D-2.12 Zinc Pellets (Arsenic Free Zinc Pellets) – AR grade.

D-2.13 Preparation of Arsenic Solution

Dissolve 0.416 g of sodium Arsenate ($Na_2HAsO_4.7H_2O$) in 1000 ml distilled water in volumetric flask after addition of 1 ml of concentrated hydrochloric acid. This is a 100 ppm arsenic solution which may be called standard *A*.

Take 1 ml of standard *A* and dilute to 100 ml in a volumetric flask. This is a 1 ppm arsenic solution which may be called Standard *B*.

D-2.14 Preparation of Working Solution

Take 5 ml, 10 ml, 15 ml, 20 ml, and 25 ml solution of Standard *B* in respective Gutzeit bottle. The solution taken in Gutzeit bottles contain 5 ppm, 10 ppm, 20 ppm, 25 ppm of arsenic respectively as shown in Fig 1 and proceed as given in **D-3** except **D-3 a)**.

D-3 PROCEDURE

a) Weigh minimum 10 g of fertilizer sample in a Gutzeit bottle.
b) Add 120 ml of water and dissolve the material.
c) Add 25 ml of 0.5 M HCl and shake.
d) Add 3 ml potassium iodide solution and wait for 5 min.
e) Add 10 ml of 0.75% stannous chloride. Add 1 g to 2 g zinc pellets and connect the Gutzeit bottle to the tube without delay, having previously placed a mercuric chloride paper in the head of the arsenic apparatus.
f) Leave for 30 min or till the reaction ceases, remove the mercuric chloride paper immediately, compare the confined spots with standard spot and ascertain the nearest concentration of Arsenic.
g) Carry out blank analysis to ensure that reagents are free from arsenic.

D-6 CALCULATION

$$\text{Arsenic (as As}_2\text{O}_3\text{), percent by mass} = \frac{Conc.\ in\ ppm \times 1.32}{Mass\ of\ the\ sample \times 10000}$$
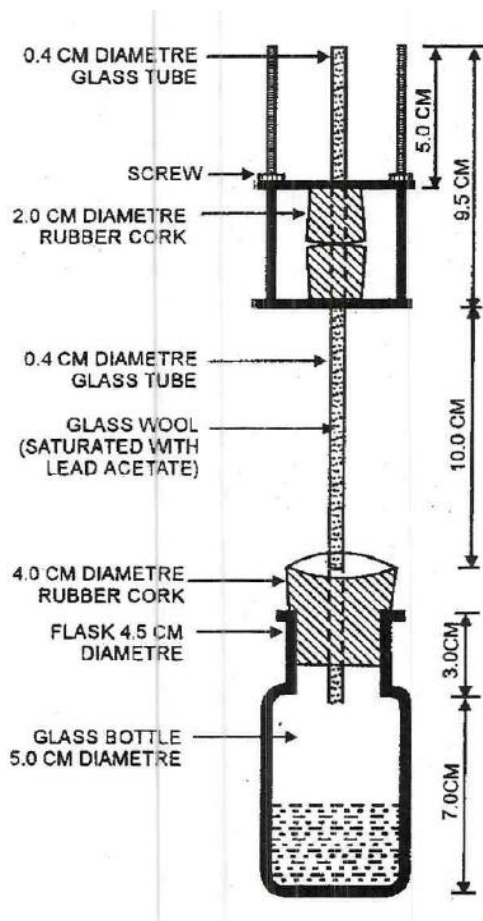

Fig. 1 MODIFIED GUTZEIT APPARATUS FOR ARSENIC ESTIMATION