भारतीय मानक **IS 14403 : 2024**

Indian Standard

मृदा संशोधन के लिए कृषि ग्रेड आयरन पाइराइट्स — विशिष्टि

(पहला पुनरीक्षण)

Agriculture Grade Iron Pyrites as Soil Amendment — Specification

(First Revision)

ICS 65.080

भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुर शाह ज़फर मार्ग, नई दिल्ली - 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI - 110002
www.bis.gov.in www.standardsbis.in

August 2024 **Price Group 5**

Soil Quality and Fertilizers Sectional Committee, FAD 07

FOREWORD

This Indian Standard (First 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.

In India, a large area of land is adversely affected by high alkali (sodic) conditions. The chemical amendments are primarily used for the reclamation of alkali soils. Gypsum has been used for a long time as a chemical amendment. The use of iron pyrites as an amendment is a recent development in the chemical amelioration of alkali soils. The use of pyrites has been found to be quite effective and has opened fresh avenues in the reclamation of alkali soils.

This standard was first published in 1996. In the preparation of this standard, considerable assistance was derived from Pyrites Phosphates and Chemicals Limited.

In this revision has been brought out to bring the Indian Standard in the latest format incorporating the latest version of the referred standard. It also incorporates one amendment issued to the previous version of this standard.

The composition of the Committee responsible for revision of the standard is given in Annex E.

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

AGRICULTURE GRADE IRON PYRITES AS SOIL AMENDMENT — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for agriculture grade iron pyrites as soil amendment.

2 REFERENCES

The standards given below contain provisions which through reference in this text, constitute provision 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 arc encouraged to investigate the possibility of applying the most recent edition of these standards:

IS No.	*Title*
IS 1070 : 2023	Reagent grade water — Specification (*fourth revision*)
IS 1288 : 1982	Methods of test for mineral gypsum (*second revision*)
IS 1289 : 1960	Methods for sampling of mineral gypsum
IS 9755 : 2021	Textiles-high density polyethylene (HDPE)/ polypropylene (pp) woven sacks for packing fertilizers — Specification (*sixth revision*)

3 TERMINOLOGY

For the purpose of this standard the following definitions shall apply.

3.1 Soil Amendment — Any substance added to the soil which improves problem soils.

3.2 Iron Pyrite — A naturally occurring sedimentary mineral (iron disulphide) which in presence of air and water forms sulphuric acid and is used as soil amendment for reclamation of alkali soil.

4 REQUIREMENTS

4.1 Sulphur Content

The sulphur content shall not be less than 16 percent by mass, when tested by the method prescribed in Annex A.

4.2 Fineness

Ninety percent of the material shall pass through a 5 mm sieve and at least 50 percent of the material shall pass through a 3 mm sieve, when tested by the method prescribed in IS 1288.

4.3 Iron Content

The iron content shall be not more than 25 percent by mass, when tested by the method prescribed in Annex B.

4.4 Aluminium Content

The aluminium content shall be not more than 10 percent by mass, when tested by the method prescribed in Annex C of the standard.

5 PACKING

The material shall be packed in HDPE bags conforming to IS 9755. The bulk manufactured material may be packed as agreed to between the purchaser and the supplier.

6 MARKING

6.1 The bags shall be marked legibly and indelibly with the following information:

a) Name of the material;

b) Name and address of the manufacturer;

c) Composition of the material (sulphur, iron and aluminum);

d) Batch number;

e) Net quantity in kg; and

f) Any other requirements as specified under the *Legal Metrology* (*Packaged Commodities*) *Rules*, 2011.

6.2 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.

6.3 Direction for use of agriculture grade iron pyrite as soil amendment shall be printed briefly on the bags as given under Annex D. A separate pamphlet may preferably be given with it.

7 SAMPLING

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

8 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 result of analysis.

ANNEX A

(*Clause* 4.1)

DETERMINATION OF SULPHUR CONTENT

A-1 PRINCIPLE

The sulphide is oxidized by bromine in carbon tetrachloride solution, followed by nitric acid. The reaction may be represented by the following equation:

$$2FeS_2 + 6HNO_3 + 15Br_2 + 16H_2O \longrightarrow 2Fe(NO_3)_3 + 4H_2SO_4 + 30HBr$$

The resultant sulphuric acid is precipitated as barium sulphate and sulphur content is determined as percentage of barium sulphate in the sample taken for the test.

A-2 REAGENTS

A-2.1 Liquid Bromine : Carbon Tetrachloride Mixture — Pure grade 2 : 3 *v*/*v*

A-2.2 Concentrated Nitric Acid — AR grade

A-2.3 Concentrated Hydrochloric Acid — AR grade

A-2.4 Whatman Accelerator or Equivalent

A-2.5 Whatman No. 540 or Equivalent Filter Paper

A-2.6 Barium Chloride — 5 percent aqueous solution (*m*/*v*)

A-3 PROCEDURE

A-3.1 Dry some finely powdered iron pyrites at 100 ºC for 1 h.

A-3.2 Weigh 0.5 g of the iron pyrites into a dry 500 ml beaker, add 6 ml of a mixture of liquid bromine and carbon tetrachloride and cover with a glass. Allow the beaker to stand in the fume cupboard for 15 min to 20 min and swirl the contents of the beaker occasionally during this period. Then add 10 ml of concentrated nitric acid gradually, and allow to stand for another 15 min to 20 min, swirling occasionally as before. Heat the covered beaker below 100 °C by placing it on a thick asbestos board over a steam bath until all action has ceased and most of the bromine has been expelled which may take about 1 h. Raise the glass cover by glass hooks resting on the rim of the beaker, or displace it to one side, and evaporate the liquid to dryness on the steam bath. Add 10 ml of concentrated hydrochloric acid, mix well, and again evaporate to dryness to eliminate most of the nitric acid. Place the beaker in an oven, maintained at 95 ºC to 100 ºC for 30 min to 60 min in order to dehydrate any silica which may be present.

NOTE — If the dry residue is heated at a temperature above 100 ºC, loss of sulphuric acid may occur and the determination will be rendered useless. Moisten the cold, dry residue with 1 ml to 2 ml of concentrated hydrochloric acid and, after an interval of 3 min to 5 min dilute with 50 ml of hot water, and rinse the sides of beaker and the cover glass with water.

Digest the contents of the beaker at 100 °C for 10 min in order to dissolve all soluble salts. Allow the solution to cool for 5 min and add 0.2 g to 0.3 g of aluminium powder to reduce the ferric iron.

Gently swirl or stir until the solution becomes colourless. Allow to cool, add a Whatman ‘accelerator’. Stir, and rinse down the cover glass and the sides of the beaker.

Filter through filter paper, and collect the filtrate in an 800 ml beaker. Wash the filter paper thoroughly with hot water. Dilute the combined filtrate and washings to 600 ml and add 2 ml of concentrated hydrochloric acid. Add from a burette, without stirring the contents of the beaker a solution of barium chloride at a rate not exceeding 5 ml per min until an excess of 5 ml to 10 ml is present. When all the barium chloride solution has been added, stir gently and allow the precipitate of barium sulphate to settle for 2 h, but preferably overnight. Filter through a filter paper or, preferably, through a porcelain filtering crucible, wash with warm water until free from chloride, and ignite to constant mass. Calculate the percentage of sulphur in the sample.

A-4 CALCULATION

$$\text{Sulphur content, percent by mass} = \frac{m}{M} \times 100$$

where

m = mass, in g, of residue left after ignition; and

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

ANNEX B

(*Clause* 4.3)

DETERMINATION OF IRON CONTENT

B-1 PRINCIPLE

Iron in pyrites in acidic medium is treated with excess ammonia to precipitate hydrous ferric oxide. Ferric oxide is converted to iron by multiplying with conversion factor 0.699 44.

B-2 REAGENTS

B-2.1 Hydrochloric Acid — 1 : 1 (*v*/*v*)

B-2.2 Concentrated Nitric Acid

B-2.3 Ammonia Solution — 1 : 1 (*v*/*v*)

B-2.4 Ammonium Nitrate — 1 percent (*m*/*v*) solution.

B-3 PROCEDURE

Weigh 0.5 g of iron pyrites into a 400 ml conical flask and add 10 ml of hydrochloric acid. Add 1 ml to 2 ml of nitric acid to the solution and boil gently until the colour is clear yellow (3 min to 5 min is usually necessary).

Dilute the solution to 200 ml with water, heat to boiling and slowly add ammonia solution in a slow stream from a small beaker until a slight excess is present, as is shown by the odour of the vapour of the above liquid. Boil the liquid gently for 1 min, and allow the precipitate to settle. The supernatant liquid should be colourless.

Soon as most of the precipitate has settled, decant the supernatant liquid through an ashless filter paper, but leave as much of the precipitate as possible in the beaker.

NOTE — It is essential that the filter paper fits the funnel properly, so that the stem of the funnel is always filled with liquid, otherwise filtration will be very slow.

Add about 100 ml of boiled ammonium nitrate solution to the precipitate, stir the mixture thoroughly and allow to settle. Decant as much liquid as possible through the filter. Wash the precipitate three to four times by decantation with 75 ml to 100 ml portions of hot ammonium nitrate solution.

Transfer the precipitate completely (and ashless filter pulp, if employed) to the filter, with the assistance of hot water from a wash bottle. Wash the precipitate several times with hot ammonium nitrate solution until it is free from chloride.

Allow each portion of the wash liquid to run through before adding the next portion and do not fill the filter more than three-fourths full of the precipitate. When the filter paper has drained thoroughly, transfer it to a previously weighed porcelain, silica or platinums crucible.

Heat the crucible gradually until content is dry. Char the paper without inflaming, and burn off the carbon at as low temperature as possible under good oxidizing conditions in order to avoid reduction of the ferric oxide. Finally, ignite the precipitate at a red heat for 15 min and take care to exclude the flame gases from the interior of the crucible, cool, in a desiccator for 15 min, and weigh. Alternatively, heat in an electric muffle furnace at 500 °C to 550 °C. Repeat the ignition (10 min to 15 min) until constant weight is obtained.

B-3 CALCULATION

$$\text{Iron content, percent by mass} = \frac{m \times 0.699\,44 \times 100}{M}$$

where

m = mass, in g, of residue left after ignition; and

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

ANNEX C

(*Clause* 4.4)

DETERMINATION OF ALUMINIUM CONTENT

C-1 PRINCIPLE

Aluminium is precipitated as the hydrated oxide by means of ammonia in the presence of ammonium chloride. The gelatinous precipitate is washed, converted into the oxide by ignition and weighed as aluminium oxide.

C-1 PROCEDURE

Weigh 1 g of iron pyrites, which is equivalent to about 0.1 g of aluminium into a 500 ml beaker, provided with a glass cover and a stirring rod. Dissolve the sample in 200 ml of water, add 5 g of ammonium chloride and a few drops of methyl red indicator in 0.2 percent alcoholic solution and heat just to boiling. Add ammonia solution (1/1) (*v*/*v*) dropwise from a burette until the colour of the solution changes to a distinct yellow. Boil the solution for 1 to 2 min, and filter at once through a suitable filter paper. Wash the precipitate thoroughly with hot 2 percent ammonium nitrate or ammonium chloride solution made neutral with ammonia solution to methyl red (or to phenol red). Place the paper with the precipitate in a previously ignited porcelain, silica or platinum crucible. Dry, char, and ignite for 10 min to 15 min with a suitable high temperature burner. Allow the crucible, covered with a well-fitting lid, to cool in a desiccator and weigh.

C-3 CALCULATION

Aluminum content, percent by mass =

$$\frac{m \times 0.529\,13 \times 100}{M}$$

where

m = mass, in g, of residue left after ignition; and

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

ANNEX D

(*Clause* 6.3)

DIRECTION FOR USE OF AGRICULTURE GRADE IRON PYRITES AS SOIL AMENDMENT

D-1 Application of pyrite be done preferably in summer to take advantage of higher temperatures for more effective oxidation.

D-2 Field should be properly prepared and given a light irrigation, to bring the soil moisture content to its near field capacity.

D-3 On moist soil surface, pyrite be dressed as uniformly and as thinly as possible, to ensure maximum availability of air to facilitate oxidation.

D-4 Sufficient time be allowed for pyrite to oxidize on the surface of the soil (7 days to 15 days).

D-5 The field should afterwards be sub-divided into small compartments and filled with irrigation water to facilitate leaching of soluble salts.

ANNEX E

(*Foreword*)

COMMITTEE COMPOSITION

Soil Quality and Fertilizers Sectional Committee, FAD 07

Organization	*Representative(s)*
Natural Resource Management, New Delhi	Dr Suresh Kumar Chaudhari (***Chairperson***)
Bidhan Chandra Krishi Vishwavidyalaya, West Bengal	Prof B. Mandal
Central Fertilizers Quality Control and Training Institute, Faridabad	Shri Shyam Babu
Consumer Guidance Society of India, Mumbai	Dr Sitaram Dixit
CSIR - National Environmental Engineering Research Institute, Nagpur	Dr Hemant J. Purohit Dr B. K. Sarangi (*Alternate*)
ICAR - Central Soil Salinity Research Institute, Karnal	Dr A. K. Rai Dr Ashim Datta (*Alternate*)
ICAR - National Bureau of Agriculturally Important Microorganisms, Kushmaur	Dr Alok kumar Srivastava Dr Hillol Chakdar (*Alternate*)
ICAR - National Bureau of Soil Survey and Land Use Planning, Nagpur	Shri Hrittick Biswas
Indian Agricultural Research Institute Library, New Delhi	Head (Soil Science)
Indian Farmers Fertiliser Cooperative, New Delhi	Dr Tarunendu Singh
Indian Institute of Soil Science, Bhopal	Dr S. P. Datta Dr S. K. Behera
Indian Micro Fertilizers Manufacturers Association, Pune	Dr Rahul Mirchandani Dr R. K. Tewatia (*Alternate*)
Insecticides (India) Limited, Delhi	Dr Lokesh Chander Rohela Dr Ritika Pathak (*Alternate*)
Institute of Agricultural Sciences, Banaras Hindu University, Varanasi	Shri Satish Kumar Singh Dr Amitav Rakshit (*Alternate*)
International Zinc Association, New Delhi	Dr Soumitra Das Shri Rahul Sharma (*Alternate*)
Mahatma Phule Krishi Vidyapeeth, Rahuri	Dr B. M. Kamble Dr A. G. Durgude (*Alternate*)
Ministry of Chemicals and Fertilizers, New Delhi	Shri Balvinder Kumar
Multiplex Bio-Tech Private Limited, Bengaluru	Shri Mahesh G. Shetty
National Centre for Organic and Natural Farming, Ghaziabad	Dr Gagnesh Sharma Shri Ravindra Kumar (*Alternate*)
National Fertilizers Limited, Noida	Dr Jyoti Goel Ms Manisha (*Alternate*)
Tamil Nadu Agricultural University, Coimbatore	Dr P. Balasubramaniam Dr K.M. Sellamuthu

Organization	*Representative(s)*
BIS Directorate General	Shrimati Suneeti Toteja, Scientist ‘F’/Director and Head (Food and Agriculture) [Representing Director General (*Ex-officio*)]

Member Secretary
Shri Kuldeep Mittal
Scientist ‘B’/Assistant Director
(Food and Agriculture), BIS

This Indian Standard has been developed from Doc No.: FAD 07 (25165).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402 *Website*: www.bis.gov.in

Regional Offices: *Telephones*

Central : 601/A, Konnectus Tower -1, 6th Floor, DMRC Building, Bhavbhuti Marg, New Delhi 110002 — 2323 7617

Eastern : 8th Floor, Plot No 7/7 & 7/8, CP Block, Sector V, Salt Lake, Kolkata, West Bengal 700091 — 2367 0012, 2320 9474

Northern : Plot No. 4-A, Sector 27-B, Madhya Marg, Chandigarh 160019 — 265 9930

Southern : C.I.T. Campus, IV Cross Road, Taramani, Chennai 600113 — 2254 1442, 2254 1216

Western : Manakalya, 4th Floor, NTH Complex (W Sector), F-10, MIDC, Andheri (East), Mumbai 400093 — 283 25838

Branches : AHMEDABAD, BENGALURU, BHOPAL, BHUBANESHWAR, CHANDIGARH, CHENNAI, COIMBATORE, DEHRADUN, DELHI, FARIDABAD, GHAZIABAD, GUWAHATI, HARYANA (CHANDIGARH), HUBLI, HYDERABAD, JAIPUR, JAMMU, JAMSHEDPUR, KOCHI, KOLKATA, LUCKNOW, MADURAI, MUMBAI, NAGPUR, NOIDA, PARWANOO, PATNA, PUNE, RAIPUR, RAJKOT, SURAT, VIJAYAWADA.

Published by BIS, New Delhi