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<b>DRAFT FOR COMMENTS ONLY</b>	<b>Doc.: FAD 07 (25165) P</b>
<p><i>Proposed Draft Indian Standard</i></p> <p><b>AGRICULTURE GRADE IRON PYRITES AS SOIL AMENDMENT — SPECIFICATION</b></p> <p><i>(first revision of IS 14403)</i></p>	
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**Soil Quality and Fertilizers Sectional Committee, FAD 07**

**FOREWORD**

*(Formal clauses would be added later)*

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. A need was, therefore, felt to formulate Indian Standard on the subject stipulating various quality parameters for the benefit of processors and users of the product. In preparation of this standard considerable assistance has been derived from Pyrites Phosphates and Chemicals Limited.

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 agriculture grade iron pyrites as soil amendment.

## 2 REFERENCES

The Indian Standards listed 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 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated:

<i>IS No.</i>	<i>Title</i>
IS 1288 : 1982	Methods of test for mineral gypsum ( <i>second revision</i> )
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 ( <i>sixth revision</i> )

## 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 (*m/m*), when tested by the method prescribed in Annex A.

### 4.2 Fineness

90 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 (*m/m*), when tested by the method prescribed in Annex A.

#### **4.4 Aluminium Content**

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

#### **5 PACKING**

The material shall be packed in moisture proof bags as agreed to between the purchaser and the supplier. Each bag shall be securely closed.

#### **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;
- f) Any other information required under the Standards of Weights and Measures (*Packaged Commodities*) Rules, 1977; and
- g) Storage instructions worded as under:  
“STORE IN OPEN UNDER SHED”

#### **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.

**ANNEX A**  
(Clause 4.1)  
**DETERMINATION OF SULPHUR CONTENT**

**A-1 PRINCIPLE**

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



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-1 REAGENTS**

**A-1.1 Liquid Bromine : Carbon Tetrachloride Mixture** — Pure grade 2:3 v/v.

**A-1.2 Concentrated Nitric acid** — Analytical reagent grade (AR).

**A-1.3 Concentrated Hydrochloric Acid** — AR grade.

**A-1.4 Whatman Accelerator or Equivalent**

**A-1.5 Whatman No. 540 or Equivalent Filter Paper**

**A-1.6 Barium Chloride** — 5 percent aqueous solution (m/v).

**A-2 PROCEDURE**

**A-2.1** Dry some finely powdered iron pyrites at 100°C, for one hour.

**A-2.2** 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 to 20 minutes 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 to 20 minutes, 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 one hour.

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 to 100°C, for 30 to 60 minutes in order to dehydrate any silica which may-be present.

NOTE - If the dry residue is heated at a temperature above 100°C, loss of sulphuric acid may occur and the determination will be rendered useless. Moisten the cold, dry residue with 1 to 2 ml of concentrated hydrochloric acid and, after an interval of 3 to 5 minutes, 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 minutes in order to dissolve all soluble salts. Allow the solution to cool for 5 minutes, and add 0.2 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 minute until an excess of 5 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 hours, 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-3 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 to 2 ml of nitric acid to the solution and boil gently until the colour is clear yellow (3 to 5 minutes 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 minute, 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 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 platinum 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 1.5 minutes and take care to exclude the flame gases from the interior of the crucible, cool, in a desiccator for 15 minutes, and weigh. Alternatively, heat in an electric muffle furnace at 500 to 550°C. Repeat the ignition (10 to 15 minutes) until constant weight is obtained.

### **B-3 CALCULATION**

$$\text{Iron content, percent by mass} = \frac{m \times 0.69944 \times 100}{M}$$

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 minutes, 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 to 15 minutes with a suitable high temperature burner. Allow the crucible, covered with a well-fitting lid, to cool in a desiccator and weigh.

## B-3 CALCULATION

$$\text{Aluminum content, percent by mass} = \frac{m \times 0.52913 \times 100}{M}$$

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 oxidise on the surface of the soil (7 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.