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भारतीय मानक ***मसौदा***

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(*आइ एस 1251 का चौथा पुनरीक्षण*)

Draft *Indian Standard*

ZINC PHOSPHIDE, TECHNICAL — SPECIFICATION

(*Fourth Revision of IS 1251*)

ICS 65.100.01

Pesticides Sectional Committee, FAD 01 **Last date of comments:** 7 December 2024

FOREWORD

(*Formal clauses would be added later*)

Zinc phosphide, technical, is widely used as rodenticide for the control of bandicoots, rats and mice. The material acts as a stomach poison and on coming in contact with gastric juices in the stomach, produces phosphine gas which kills the rodents.

This standard was first published in 1958 and subsequently revised in 1973 and 1984. In the third revision issued in 1988, the requirements were updated.

In this fourth revision, the standard has been brought out in the latest style and format of the Indian Standards, and references to Indian Standards wherever applicable have been updated. It also incorporates one amendment issued to the previous version of this standard.

In the preparation of this standard, due consideration has been given to the provisions of the *Insecticides Act*, 1968 and rules framed thereunder. However, this standard is subject to the restriction imposed under these regulations, 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 the methods of sampling and test for zinc phosphide, technical.

2 REFERENCES

The following standards 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 indicated below:

IS No.	*Title*
IS 460 (Part 1) : 2020	Test Sieves — Specification: Part 1 Wire cloth test sieves (*fourth revision*)
IS 1070 : 2023	Reagent grade water – Specification (*fourth revision*)
IS 6940 : 202X	Pesticides and their formulations – Test methods (*second revision*) [*Under preparation Doc: FAD 01(25870)WC*]
IS 8190 (Part 1) : 1988	Requirements for packing of pesticides: Part 1 Solid pesticides (*second revision*)
IS 8190 (Part 3) : 1979	Requirements for packing of pesticides: Part 3 Household pesticides
IS 10946 : 1996	Methods of sampling for technical grade pesticides (*first revision*)

3 REQUIREMENTS

3.1 Description

The material shall be dark-grey powder with a characteristic garlic odour. It shall be fine, heavy, free flowing powder free from lumps.

3.2 The material shall also comply with the requirements given in Table 1.

3.3 Freedom from Sulphides — The material shall be free of sulphides when tested by the method prescribed in Annex C.

4 PACKING

The material shall be packed according to the requirements given in IS 8190 (Part 1) and IS 8190 (Part 3).

5 MARKING

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

a) Name of the material;
b) Name of the manufacturer;
c) Date of manufacture;
d) Batch number;

e) Nominal zinc phosphide contents; and
f) Cautionary notice as worded in the *Insecticides Act*, 1968, and Rules framed thereunder; and
g) Any other information required under the *Legal Metrology* (*Packaged Commodities*) *Rules*, 2011.

5.1 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.

Table 1 Requirements for Zinc Phosphide, Technical

(*Clause* 3.2)

SI No.	Characteristic	Requirement	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Zinc phosphide (Zn_3P_2), percent by mass, *Min*	80.0	Annex A
ii)	Zinc content, percent by mass, *Min*	60.0	Annex B
iii)	Sieving requirements (*see* Note)		
	a) Material passing through 150 micron IS Sieve [*see* IS 460 (Part 1)], *Min*	99.0	IS 6940
	b) Material passing through 106 micron IS Sieve [*see* IS 460 (Part 1)], *Min*	92.0	IS 6940

Note — For sieving requirement, start with 50 g of the material.

6 SAMPLING

Representative samples of the material shall be drawn as specified in IS 10946.

7 TEST

7.1 Tests shall be carried out in accordance with **3.3** and col 4 of Table 1.

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

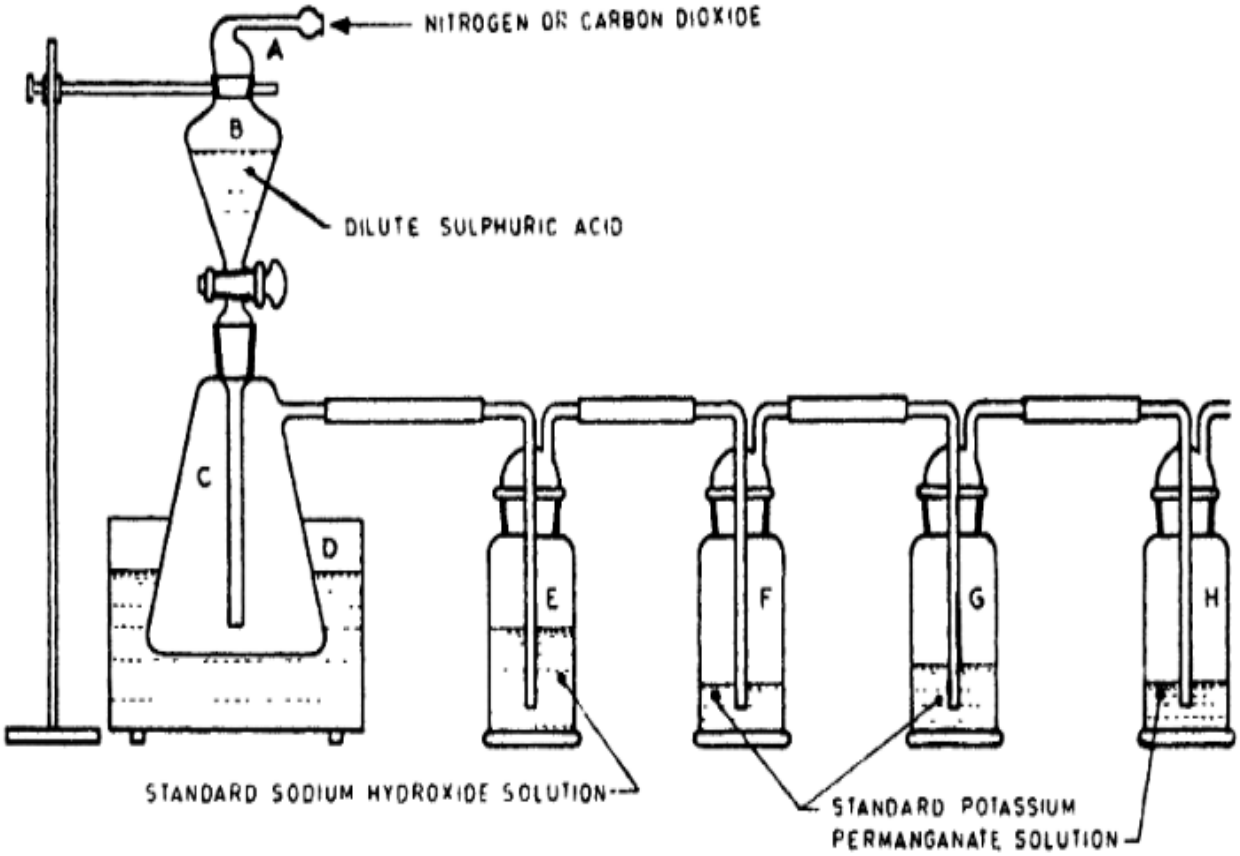
[*Table* 1, *SI No.* (i)]

DETERMINATION OF ZINC PHOSPHIDE (Zn_3P_2) CONTENT

A-1 APPARATUS

A-1.1 The assembly of the apparatus is shown in Fig. 1.

A-1.2 The apparatus consists of a 250 ml reaction flask *C* with a standard interchangeable socket. A 250 ml separating funnel *B* and delivery tube (PVC tube surgical soft type) as to be connected according to Fig. 1. The side tube is serially connected with four 200/250 ml absorption bottles *E*, *F*, *G* and *H* respectively. To the mouth of the separating funnel is attached a nitrogen or carbon dioxide inlet tube *A* as indicated in Fig. 1. The reaction flask is so mounted on a stand where it is possible to immerse it in the water bath *D* maintained at a temperature of (65 ± 5) °C.



A = Adapter tubing (for nitrogen or carbon dioxide gas)

B = Separating funnel conical shape with cone and socket 250 ml

C = Reaction flask (Buchner) 250 ml

D = Thermostatically controlled water — bath

E =
F = } Gas wash (absorption) bottles (200/260 ml)
G =
H =

FIG. 1 ASSEMBLY OF APPARATUS FOR THE DETERMINATION OF ZINC PHOSPHIDE AND DETECTION OF SULPHIDES

A-2 REAGENTS

A-2.1 Standard Potassium Permanganate Solution — Approximately 0.5 N.

A-2.2 Sulphuric Acid — 10 percent (*m*/*v*)

A-2.3 Nitrogen Gas or Carbon Dioxide Gas – From a cylinder

A-2.4 Sulphuric Acid — 1:1 (*v*/*v*)

A-2.5 Standard Sodium Hydroxide Solution — 1 N.

A-2.6 Standard Oxalic Acid Solution — Approximately 0.5 N and acidified with sulphuric acid. Weigh accurately about 15.8 g of oxalic acid ($H_2C_2O_4.2H_2O$) and dissolve in about 200 ml of water contained in a 500 ml volumetric flask. Add to the volumetric flask 125 ml of sulphuric acid 1:1 (*v*/*v*), make up the volume with water and mix.

A-3. Procedure

A-3.1 Measure 100 ml of standard sodium hydroxide solution into the gas wash bottle (absorption bottle) *E*, 100 ml of standard potassium permanganate solution in gas wash bottle. Measure 50 ml of standard potassium permanganate solution into each of the gas wash bottles *G* and *H*. Assemble the apparatus as shown in Fig. 1 (without dilute sulphuric acid in the separating funnel B). Pass the gas (nitrogen or carbon dioxide) slowly through the apparatus so as to displace air. Weigh accurately about 0.5 g of the material and transfer it into the reaction flask *C* quantitatively.

A-3.2 Disconnect the nitrogen gas tube and place 100 ml of dilute sulphuric acid in the separating funnel *B*. Connect the nitrogen gas tube to the funnel again. Add dilute sulphuric acid (*see* **A-2.2**) to the reaction flask slowly and very cautiously drop by drop in the beginning, carefully regulating the rate of addition in such a way that a steady stream of bubbles appears in the gas wash bottles. When the addition of dilute sulphuric acid is complete, adjust the pressure of nitrogen gas or carbon dioxide gas so that a steady flow of bubbles is maintained in the reaction flask and the gas wash bottles. During this process, immerse the reaction flask *C* in the water bath maintained at a temperature of (65 ± 5) °C. Continue the reaction for at least 90 min. Sweep the last traces of

phosphine from the flask with more rapid stream of nitrogen or carbon dioxide for at least 5 minutes. At the end of the reaction and sweeping period, disconnect the apparatus and quantitatively transfer the redused potassium permanganate solution contained in the three gas wash bottles *F*, *G* and *H* to a 1000 ml or a convenient size beaker. Rinse the gas wash bottles and connecting tubes with 200 ml of standard oxalic acid solution, taking care to dissolve all the manganese dioxide. Add the rinsings to the reduced potassium permanganate solution contained in the beaker. Rinse the gas wash bottles and connecting tubes with water and transfer the rinsings to the same beaker. Warm the contents of the beaker to approximately 60 °C and titrate the excess oxalic acid with standard potassium permanganate solution.

A-3.3 Retain flask *C* for zinc determination as prescribed in Annex B and gas wash bottle *E* for detection of sulphides as prescribed in Annex C.

A-4 Calculation

$$\text{Zinc phosphide } (Zn_3P_2) \text{ content, percent by mass} = \frac{1.613[(200+A)N_1 - 200N_2]}{M}$$

where

A = volume, in ml, of standard potassium permanganate solution required for the titration of excess oxalic acid;

N_1 = normality of standard potassium permanganate solution;

N_2 = normality of standard oxalic acid solution; and

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

ANNEX B

[*Table* 1, *SI No.* (ii)]

DETERMINATION OF ZINC CONTENT

B-1 REAGENT

B-1.1 Sodium Hydroxide — 1 N

B-1.2 Disodium Ethylenediamine Tetra-Acetate (EDTA) Solution — 0.1 N

Dissolve 18.6 g of disodium ethylenediamine tetraacetate dihydrate in water and make up the volume to 1 litre in a volumetric flask. Standardize according to the procedure given in **B-2**.

B-1.3 Standard Zinc Solution — Dissolve 3.269 g of analytical reagent grade zinc metal in 20 ml of hydrochloric acid (1:1 *v*/*v*) and make up to 1000 ml in a volumetric flask

B-1.4 Erichrome Black T-Indicator chloride — Mix thoroughly 1 g of Erichrome black T with 100 g of sodium chloride.

B-1.5 Buffer Solution — Dissolve 67.5 g ammonia solution (570 ml) and make it to of ammonium chloride in 300 ml water, add strong 1 000 ml with distilled water.

B-1.6 Sodium Hydroxide — 0.5 N

B-1.7 Sulphuric Acid — 10 percent (*v*/*v*)

B-1.8 Ammonia Solution — 10 percent (*v*/*v*)

B-2 Standardization of EDTA Solution — Pipette out 25 ml of standard zinc solution **(B-1.3)** in a 250 ml Erlenmeyer flask. Neutralize it with 1 N sodium hydroxide. Add 5 ml of a buffer solution (*see* **B-1.5**) and few specks of Erichrome black T. Titrate with EDTA solution. The end point is indicated by a sharp colour change from red to blue.

One ml of 0.1 N EDTA solution = 3.269 mg of pure zinc.

B-3 Procedure

Filter retained solution (*see* **A-3.3**) and make up to 250 ml in standard volumetric flask. Take 25 ml aliquot in 500 ml Erlenmeyer flask. Add 1 N sodium hydroxide till brown precipitate of iron is complete. Then add 25 ml of sodium hydroxide. Filter the solution and wash the precipitate with dilute sodium hydroxide solution (10 ml) twice. Wash with distilled water (10 ml). Make filtrate solution neutral to litmus paper with dilute sulphuric acid, make it alkaline with dilute ammonia (*see* **B-1.8**) and add 10 ml buffer solution. Titrate with EDTA solution using Erichrome black T indicator (few specks) till clear blue end point.

B-4 Calculation

$$\text{Zinc content, percent by mass} = \frac{32.69 \times V \times N}{M}$$

Where

V = volume, in ml, of EDTA solution required for 25 ml aliquot in **B-3**;
N = normality of EDTA; and
M = mass, in g, of the material taken for test (*see* **A-3.1**)

ANNEX C
(*Clause* 3.3)
DETECTION OF SULPHIDES

C-1. REAGENT

C-1.1 Standard Neutral Cadmium Sulphate Solution — 2 M

C-2 PROCEDURE

At the end of the reaction and sweeping, disconnect gas wash bottle *E* (*see* **A-3.2** and **A-3.3**). Add 10 ml of standard neutral cadmium sulphate solution (*see* **C-1.1**) into the gas wash bottle *E*. No

yellow precipitate shall be formed. Formation of yellow precipitate indicates presence of sulphide (cadmium sulphide).