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Draft Indian Standard

Zinc Phosphate Pigment for Paints — Specification

(First Revision of IS 10897)

(ICS 71.060)

Raw materials for Paints, Varnishes and Related
Product Sectional Committee, CHD 21

Last Date for Comments: 22nd October 2025

FOREWORD

(Formal clause will be added later)

Conventionally, zinc chromate pigment has been extensively used as a corrosion-inhibitive pigment in the manufacture of ready-mixed paints and primers. However, its major drawback lies in its toxicity, as it contains hexavalent chromium (Cr^{6+}), which is both carcinogenic and harmful to the environment. In contrast, zinc phosphate has emerged as a safer and eco-friendly alternative. It provides effective corrosion protection by forming a passive phosphate layer on the metal surface while also enhancing the adhesion of the coating. Furthermore, zinc phosphate is compatible with a wide range of binder systems and is now the preferred choice for modern protective coatings due to its balance of effectiveness, and safety.

Zinc phosphate pigment exists in three types which depend on the water of crystallization content in the pigment. Type I consists predominantly of zinc phosphate dihydrate and exhibits a loss of ignition between 8.5 and 10 percent by mass at 600 °C. Type 2 consists essentially of a mixture of zinc phosphate dihydrate and zinc phosphate tetrahydrate and exhibits a loss of ignition between 10 and 13 percent by mass at 600 °C. Type 3 consists predominantly of zinc Phosphate tetrahydrate and exhibiting a loss on ignition between 13 and 16 percent by mass at 600 °C.

Zinc phosphate pigment currently manufactured in India belong to Type 1 and consists essentially of zinc phosphate dihydrate. However, zinc phosphate dihydrate pigment has got a tendency to get slowly converted into tetrahydrate type on storage. Therefore, after some time, pigment exists in the mixture of two types.

This standard was first published in 1984 and in the preparation of this standard, assistance was derived from BSI 5193 : 1975, Specification for zinc phosphate¹, issued by the British Standards Institution (BSI).

This first revision has been undertaken to align the product standards with current market demands, enhancing its acceptance and relevance. The following changes have been made:

- Zinc phosphate has been categorized into three types based on the water of crystallization content in the pigment;
- To address health and safety concerns, restrictions for lead has been tightened;
- A suitable precautionary note has been added in the marking clause in order to prevent unforeseen events, and;
- Additionally, the various editorial corrections, and references have been updated to ensure accuracy and relevance in the revised standards.

The packing of the material (*see 5.1*) and requirement of bulk density/ tapped volume calls for an agreement between the purchaser and the supplier.

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.

*Draft Indian Standard***ZINC PHOSPHATE PIGMENT FOR PAINTS — SPECIFICATION***(First Revision)***1 SCOPE**

This standard prescribes requirements and methods of sampling and tests for zinc phosphate intended for use as a corrosion inhibitive pigment in paint formulations.

2 REFERENCES

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

<i>IS No.</i>	<i>Title</i>
IS 33 : 1992	Inorganic pigments and extenders for paints - Methods of sampling and test (<i>third revision</i>)
IS 1070 : 2023	Reagent grade water — Specification (<i>fourth revision</i>)
IS 1303 : 2024	Paints - Glossary of Terms (<i>third revision</i>)

2 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 33 and IS 1303 shall apply.

3 TYPES

In this standard, zinc phosphate pigments are classified as one of the following three types:

- Type 1** — Consisting predominantly of zinc phosphate dihydrate and exhibiting a loss on ignition at 600 °C of greater than 8.5 percent (*m/m*) but less than or equal to 10.0 percent (*m/m*);
- Type 2** — Consisting essentially of a mixture of zinc Phosphate dihydrate and zinc Phosphate tetrahydrate and exhibiting a loss on ignition at 600 °C of greater than 10.0 percent (*m/m*) but less than or equal to 13.0 percent (*m/m*); and
- Type 3** — Consisting predominantly of zinc Phosphate tetrahydrate and exhibiting a loss on ignition at 600 °C of greater than 13.0 percent (*m/m*) but less than or equal to 16.0 percent (*m/m*).

4 REQUIREMENTS**4.1 Form and Condition**

The material shall be in the form of a fine white powder or in such a condition that it can be reduced to the powder form by crushing without grinding action, under a palette knife.

4.2 Composition

4.2.1 A white corrosion-inhibiting pigment consisting either predominantly of zinc phosphate dihydrate $[\text{Zn}_3(\text{PO}_4)_2 \cdot 2\text{H}_2\text{O}]$ or of a mixture of Zinc Phosphate dihydrate and zinc phosphate tetrahydrate $[\text{Zn}_3(\text{PO}_4)_2 \cdot 4\text{H}_2\text{O}]$ or predominantly of zinc phosphate tetrahydrate, and free from extenders and other pigments.

4.2.2 The material shall be free from visible impurities. It shall also be free from organic dyestuffs or substrate of any kind. It shall be free from extenders and diluents.

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

4.4 When lead free zinc phosphate pigment is required, it shall contain not more than 90 ppm of lead or compounds of lead (calculated as metallic lead), when tested by the method specified in IS 33.

5 PACKING AND MARKING**5.1 Packing**

The material shall be packed in suitable containers as agreed to between the purchaser and the supplier.

5.2 Marking

The containers shall be marked with the following particulars:

- a) Name of the material;
- b) Manufacturer's name and his recognized trade-mark, if any;
- c) Mass of the material;
- d) Type and composition of material;
- e) Declared shelf life;
- f) Batch number or lot number in code or otherwise; and
- g) Month and year of manufacture.

5.2.1 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.

6 SAMPLING

6.1 Representative samples of the material shall be drawn as prescribed in IS 33.

6.2 Criteria for Conformity

A lot shall be declared as conforming to the requirements of this standard if the test results of the composite sample satisfy the requirements prescribed in 4.

7. TEST METHODS

7.1 Test shall be conducted as prescribed in IS 33 and in Annexes A to D mentioned in col (4) of Table 1.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the result of analysis.

Table 1 Requirements for Zinc Phosphate Pigment For Paints

(Clause 4.3 and 7.1)

Sl No.	Characteristic	Requirements	Method of Test, Ref To
(1)	(2)	(3)	(4)
i)	Colour	Close match to the approved sample	—
ii)	Loss on ignition, percent by mass (<i>see</i> Note)		Annex A
	Type 1	8.5 – 10	
	Type 2	10 - 13	
	Type 3	13 - 16	
iii)	Zinc content (as Zn) calculated on ignited pigment, percent by mass	50.2 to 52	Annex B
iv)	Phosphate content (as PO ₄), on ignited pigment, percent	47 – 49.2	Annex B

	by mass		
v)	Specific resistance of aqueous extract, in ohms, <i>Min</i>	30 000	Annex C
vi)	Volatile matter, percent by mass, <i>Max</i>	1.0	IS 33
vii)	Residue on sieve, percent by mass, <i>Max</i>	0.5	IS 33
viii)	Oil absorption, m/m	19 ± 2 or it shall be within ± 10 percent of the approved sample/ declared value of the manufacturer/ as agreed between the parties	IS 33
ix)	Relative density† at 25/25°C	3.0 to 3.6	IS 33
x)	pH of aqueous extract	6 to 8	IS 33
xi)	Sum of chloride content (as Cl) and sulphate content (as SO ₄) percent by mass, <i>Max</i>	0.01	Annex D
NOTE — As zinc phosphate pigment has got a tendency to get converted from dihydrate to tetrahydrate, higher loss of ignition up to 16 percent by mass should not be a cause of rejection.			
*Methods of sampling and test for inorganic pigments and extenders for paint (<i>second revision</i>).			
†Synonymous with specific gravity.			

ANNEX A

[Clause 7.1, Table 1, Sl. No.(ii)]

DETERMINATION OF LOSS ON IGNITION

A-1 APPARATUS

A-1.1 Silica Crucible

A-1.2 Desiccator

A-2 PROCEDURE

A-2.1 Weigh about 2 to 3 g of the pigment, to the nearest 0.1 mg into a previously ignited and tared silica crucible. Heat the crucible and its content to dull red heat (500 °C to 600 °C) in air for 30 min. Cool in a desiccator and weigh. Repeat heating, cooling and weighing procedure until constant mass is obtained.

A-3 CALCULATION

A-3.1 Calculate the loss of mass of pigment on ignition as a percentage as follows:

$$\text{Loss in ignition, percent by mass} = \frac{100(M_1 - M_2)}{M_1}$$

where

M_1 = mass of the pigment taken for the test in g; and

M_2 = mass of the residue after ignition in g.

ANNEX B

[Clause 7.1, Table 1, Items (iii) and (iv)]

DETERMINATION OF ZINC AND PHOSPHATE CONTENTS

B-1 REAGENTS

B-1.1 Citromolybdate Reagents

B-1.1.1 Dissolve 70 g of sodium molybdate dihydrate, ($\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$) in 150 ml of water and mark it as Solution No. 1.

B-1.1.2 Add 85 ml of nitric acid (relative density 1.48) to a solution of 60 g of citric acid monohydrate ($\text{C}_6\text{H}_8\text{O}_7 \cdot \text{H}_2\text{O}$) in 150 ml of water and mark it as Solution No. 2.

B-1.1.3 Mix the Solution No. 1 with the Solution No. 2 with constant stirring and mark it as Solution No. 3.

B-1.1.4 Add 35 ml of nitric acid (relative density 1.48) to 100 ml of water, then add 5 ml of pure freshly distilled quinoline with stirring and mark it as Solution No. 4.

B-1.1.5 Pour the Solution No. 4 into the Solution No. 3 stir and allow to stand for 12 h. Then filter through a sintered glass filter crucible, porosity Grade 4 μm to 16 μm . Store the solution in a tightly closed bottle, protected from light. Add 280 ml of acetone to the solution and dilute with water to 1 litre. Store this solution not longer than one week, in a tightly closed bottle and protect from light.

B-1.2 EDTA solution (0.01 M)

Dissolve 3.7225 g of disodium ethylenediamine tetracetate dihydrate (EDTA disodium salt) in water in a 1000 ml volumetric flask, dilute to the mark and mix.

B-1.3 Xylenol Orange Mixture

Mix well 1 g of xylenol orange with 100 g of sodium chloride.

B-1.4 Hexamethylene Tetramine

B-1.5 Dilute Hydrochloric Acid

Dilute 10 ml of hydrochloric acid (relative density 1.19) with 40 ml of water.

B-2 PREPARATION OF THE TEST SOLUTION

Weigh to the nearest 0.1 mg of a test portion of approximately 1 g, previously ignited as described in 7 of IS 33 and boil with 50 ml of dilute hydrochloric acid for 20 minutes. After cooling down, transfer the solution into a 1 000 ml volumetric flask, dilute to 1 000 ml and mix well (test solution).

B-3 DETERMINATION OF ZINC CONTENT**B-3.1 Procedure**

B-3.1.1 Pipette 50 ml of the test solution into a 150~ ml beaker. After addition of 0.1 g of xylenol orange mixture, add small portions of hexamethylene tetramine until the colour changes from yellow to pink. Then add an excess of 0.4 to 0.5 g hexamethylene tetramine. Afterwards, while stirring, titrate with the EDTA solution until the colour changes to yellow.

B-3.1.2 Blank Test

Carry out a blank test in the same way as the determination, but omitting test portion.

B-3.2 Calculation — Calculate zinc content of the sample, as follows:

$$\text{Zinc content (as Zn), percent by mass} = \frac{(V_1 - V_0) \times 1.3074}{m}$$

where

V_0 = volume of EDTA solution, required in the blank test in ml;

V_1 = volume in ml of EDTA solution, required for the sample in ml;

m = mass of the test sample in g.

B-4 DETERMINATION OF PHOSPHATE CONTENT

B-4.1 Two methods have been prescribed for the determination of phosphate content. *Method 1* gives a volumetric method and *Method 2* gives a gravimetric method. The volumetric method shall be used for routine analysis and the gravimetric method shall be used as a reference method.

B-4.2 Method 1 — Volumetric Method (by Ion Exchange)**B-4.2.1 Outline of the Method**

Paint pigments containing zinc phosphate are shaken with a strong cation exchange resin. The resulting phosphoric acid is titrated with standard alkali using a mixed indicator solution.

B-4.2.2 Principle

Strong cation exchange resins in the hydrogen form are capable of solubilising zinc phosphate by ion exchange in the presence of water. The trivalent phosphoric acid formed can be treated as a mixture of three monovalent acids with dissociation constants $K_1 = 7.5 \times 10^{-3}$ (pH 4.6) $K_2 = 6.2 \times 10^{-8}$ (pH 9.7) and $K_3 = 5 \times 10^{-13}$ (no specific pH). Pigments other than zinc phosphate could possibly give rise to some acidic products which would be included in the titration to reach K_1 . Consequently the phosphate content is calculated from the titration volume between K_1 and K_2 . A mixed indicator solution is used to identify the equivalence points.

B-4.2.3 Apparatus**B-4.2.3.1 Mechanical flask shaker.****B-4.2.4 Reagents****B-4.2.4.1 Strong cation exchange resin in the hydrogen form.**

Type: Sulphonated polystyrene resin cross linked with divinylbenzene in bead form approx. 14-52 mesh.

NOTE — These resins are amberlite — IR 120; zeolite — 325 and dowex — 50W.X8.

Regeneration of ion exchange resin may be done as follows:

Transfer the resin and insoluble pigments from the filter to a clean 250 microns IS Sieve and wash well with water to remove the pigments through the sieve. Soak the separated resin for 24 hours (with occasional stirring) in 10 percent hydrochloric acid prepared by adding 1 part concentrated hydrochloric acid to 2 parts water by volume.

After regeneration, wash the resin acid-free by decantation. It is recommended that the resin be shaken with water for several hours, decanting the water at 30 min intervals and topping up with clean water.

B-4.2.4.2

Mixed indicator solution	m/v
Methyl red	0.02
Bromocresol green	0.06
<i>O</i> — Cresolphthalein	0.24
Metanil yellow	0.03
Xylene cyanol FF	0.01

Method of preparation:

- Step 1 Dissolve with warming 0.06 g bromocresol green, 0.24 g o-cresolphthalein and 0.03 g metanil yellow in 50 ml of 1:1 v/v industrial methylated spirits and water. When the solution is complete, add 0.01 g xylene cyanol FF to the warm solution and swirl to dissolve. Mark the solution as (A).
- Step 2 Dissolve with warming, 0.02 g of methyl red in 30 ml of acetone and mark the solution as (B).
- Step 3 Mix solution A and B and make up to 100 ml with water.

B-4.2.4.3 Standard sodium hydroxide solution — 0.1 M.**B-4.2.5 Procedure**

Accurately weigh (M_1) sufficiently dried pigment to contain approximately 0.4 g of zinc phosphate dihydrate into a 250 ml stoppered flask. Add 20 g of wet cation exchange resin and 100 ml of water. Stopper the flask and shake for 1 hour on a mechanical shaker. Separate the resin and any insoluble pigments by quantitatively transferring the contents of the flask to a glass fiber funnel containing a glass wool plug. Wash with 25 ml portions of water until the washings are free from acid. Collect the filtrate and washings in a 250-ml conical flask. Add 8 drops of the mixed indicator solution and titrate with the standardized sodium hydroxide solution until the first hydrogen end point is reached. Colour changes from red through very pale red to colourless. Note the titre (T_1). Continue the titration to the second hydrogen end point; colour changes from blue to green through blue to violet blue. Note the titre (T_2).

B-4.2.5.1 Carry out a blank test in the same way as the determination, but omitting the test portion. Note the titre (T_3) for the first end point and (T_4) for the second end point.

B-4.2.5.2 Calculation

Calculate the phosphate content as follows:

$$\text{Phosphate content, percent by mass} = \frac{\{(T_2 - T_1) - (T_4 - T_3)\} \times M \times 9.497}{M_1}$$

where

M = molarity of the sodium hydroxide solution; and

M_1 = mass of the material taken for the test in g.

B-4.3 Method 2 — Gravimetric Method

B-4.3.1 Procedure — Pipette 50 ml of the test solution prepared as in **B-2** into a 400 ml beaker, dilute with 50 ml of water and add 100 ml of the citromolybdate reagent. Cover the beaker with a watch glass and heat on a hot plate for 30 seconds to $(7.5 \pm 5)^\circ\text{C}$. Do not use a flame for heating and do not stir, neither during addition of the reagent nor during heating in order to avoid any formation of lumps. After cooling down to room temperature, stir 3 to 4 times using a glass rod. Heat a sintered glass filter crucible, porosity 4 to 16 pm, at $(250 \pm 10)^\circ\text{C}$ for 15 minutes. Cool down to room temperature in a desiccator and weigh it to the nearest 0.1 mg. Filter the liquid through the sintered glass crucible and wash the residue six times with 30 ml portions of water. Transfer the residues to the crucible and wash it again four times with water. Suck off the washing water by means of a vacuum pump. Heat the filter crucible for 15 minutes in an oven maintained at $(250 \pm 10)^\circ\text{C}$. After cooling down to room temperature, weigh the crucible to the nearest 0.1 mg. Carry out a blank test in the same way as the determination, but omitting the test portion.

B-4.3.2 Calculation

Calculate the phosphate content of the sample as follows:

$$\text{Phosphate content, percent by mass} = \frac{(m_1 - m_0) \times 85.829}{m}$$

where

m = mass, of the test sample in g;

m_0 = mass, of the residue obtained in the blank test in g; and

m_1 = mass, of the residue obtained in the sample in g.

ANNEX C

[Clause 7.1, Table 1, Item (v)]

DETERMINATION OF SPECIFIC RESISTANCE OF AQUEOUS EXTRACT**C-1 REAGENTS**

C-1.1 Conductivity Water — Resistivity not less than 250 000 ohms at 23 °C.

C-1.2 Methanol — Resistivity not less than 250 000 ohms at 23 °C.

C-1.3 Potassium Chloride — 0.02 M solution.

C-2 APPARATUS

C-2.1 Centrifuge or Ultra Centrifuge

C-2.2 Filter Paper

Fine textured which shall be washed with conductivity water on a filter funnel until the filtrate gives a specific resistance of 200 000 ohms at 23 °C.

C-2.3 Conductivity Bridge

C-2.4 Conductivity Cell — having a cell constant K of approximately 1.

C-3 DETERMINATION OF CELL CONSTANT

Prepare a working standard solution of potassium chloride by diluting the 0.02 M solution with conductivity water to a known concentration. Measure the resistance R of this solution using the conductivity cell at 23 °C. Calculate the cell constant by the formula:

$$K_{23} = \frac{R}{P}$$

where

R = measured resistance in ohms, and

P = specific resistance at 23 °C of potassium chloride solution of the concentration used (for an 0.000 2 M solution, this is 3 440 ohms).

C-4 PROCEDURE

Add 20 g \pm 0.01 g of the material and 180 g of boiling conductivity water in a tared beaker. Boil for 5 min with occasional stirring, cool to 60 °C and bring the net mass to 200 g. Filter through fine textured filter paper, reject first 10 ml, cool at 23 °C and take the readings.

C-5 EXPRESSION OF RESULTS

Calculate the resistivity, P_{23} in ohms, of the aqueous extract of the pigment at 23 °C by means of the formula:

$$P_{23} = \frac{R_{23}}{K_{23}}$$

where

R_{23} = mean of the measured values of resistance in ohms, and

K_{23} = cell constant.

ANNEX D

[Clause 7.1, Table 1, Item (xi)]

DETERMINATION OF WATER SOLUBLE CHLORIDES AND SULPHATES

D-1 REAGENTS

D-1.1 Hydrochloric Acid — relative density 1.18

D-1.2 Silver Nitrate — 0.01 N

D-1.3 Barium Chloride — 50 g/l

D-1.4 Potassium Chromate — 50 g/l

D-2 APPARATUS

D-2.1 Sintered Silica Crucible — Porosity 4 µm to 16 µm.

D-3 PREPARATION OF THE AQUEOUS EXTRACT

To a 20 g ± 0.01 g of the sample, add 180 g of boiling conductivity water in a tared beaker. Boil for 5 min with occasional stirring. Cool to 60 °C and bring the net mass to 200 g. Filter through a fine textured filter paper. Reject the first 10 ml. Cool and preserve the filtrate for the determination of sulphates and chlorides.

D-4 PROCEDURE

D-4.1 Determination of Sulphates

Take 50 ml of the aqueous extract as obtained in **D-3**, acidify with 3 ml of hydrochloric acid and boil vigorously. Add the barium chloride solution drop by drop to the hot solution until in slight excess and allow the solution to stand overnight. Decant the supernatant liquid through the sintered tared silica crucible, transfer the precipitate to the crucible, wash it free from chlorides, ignite it gently and then at red heat, cool in a desiccator and weigh to the nearest milligram.

D-4.1.1 Expression of Results

Calculate the water soluble sulphates content, (expressed as SO_4), as a percentage of mass by the following formula:

$$\frac{165 m_1}{m_0}$$

where

m_0 = mass of the pigment in g; and

m_1 = mass of barium sulphate in g.

D-4.2 Determination of Chlorides

Take 50 ml of the aqueous extract as obtained in **D-3**, add 1 ml of potassium chromate solution and titrate with standard silver nitrate solution with vigorous shaking until a faint reddish brown colour persists. Carry out a blank determination by adding 1 ml of potassium chromate to 50 ml of water and titrate with silver nitrate solution till the colour matches that of the previous titration making due allowance for opalescence or turbidity.

D-4.2.1 Expression of Results

Calculate water soluble chlorides (expressed as Cl) as percentage of mass by the formula:

$$\frac{0.1418(V_1 - V_0)}{M}$$

where

V_1 = volume of silver nitrate solution used for titration of sample in ml;

V_0 = volume of silver nitrate solution used in blank in ml; and

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