IS 35 (1975): Zinc oxide for paints [CHD 20: Paints, Varnishes and Related Products]

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Indian Standard
SPECIFICATION FOR ZINC OXIDE FOR PAINTS
(First Revision)

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 3

May 1976

AMENDMENT NO. 1 OCTOBER 1987

TO

IS : 35-1975 SPECIFICATION FOR ZINC OXIDE FOR PAINTS

(First Revision)

[Page 4, Table 1, Sl No, (iii), col 2]:

a) Add the symbol '+' after oil absorption.

b) Add the following foot-note at the end of the tables'

'+This shall, however, be within ±10 percent of the approved sample, if any.'

(CDC 50)

Reprography Unit, BIS, New Delhi, India

Indian Standard

SPECIFICATION FOR ZINC OXIDE FOR PAINTS

(First Revision)

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Indian Standard

SPECIFICATION FOR ZINC OXIDE FOR PAINTS

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 15 December 1975, after the draft finalized by the Raw Materials for Paint Industry Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard was first published in 1950 and was largely based on the interim co-ordinated draft produced with the assistance of representatives of manufacturers and various departments and authorities of the Government of India by the Co-ordinating Subcommittee of the No. 5 Standing Committee on Specifications for Paints and Allied Stores of the General Headquarters India (now Army Headquarters).

0.3 In this revision, requirement for oil absorption has been modified and an additional requirement for sulphur content has been included. For estimation of zinc oxide content EDTA method has been prescribed in place of the gravimetric method and the volumetric method has been adopted from ISO/R 275-1962 'Zinc oxide for paints' issued by the International Organization for Standardization (ISO) with Type I of which this standard is substantially aligned.

0.4 This standard contains clause **4.1** which calls for agreement between the purchaser and the supplier.

0.5 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: 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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1. SCOPE

1.1 This standard prescribes requirements and methods of sampling and test for zinc oxide pigment for paints.

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*Rules for rounding off numerical values (*revised*).

2. TERMINOLOGY

2.1 For the purpose of this standard, the definitions given under 2 of IS : 33-1963* and in IS : 1303-1963†, shall apply.

3. REQUIREMENTS

3.1 Form and Condition — The material shall be in the form of fine dry powder free from grit or in such a condition that it can be readily reduced to the powder form by crushing under a palette-knife without any grinding action.

3.2 Composition — The pigment, dried as described under 6 of IS : 33-1963*, shall consist of not less than 99.0 percent of zinc oxide when determined by the method given in Appendix A.

3.3 Lead-Free Material — When lead-free zinc oxide is required, it shall contain not more than 0.03 percent of lead or compounds of lead (calculated as metallic lead) and tested by the method specified under **19** of IS : 33-1963*.

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

TABLE 1 REQUIREMENTS FOR ZINC OXIDE FOR PAINTS

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO CL NO. IN	
			Appendix	IS : 33-1963*
(1)	(2)	(3)	(4)	(5)
i)	Volatile matter, percent by mass, *Max*	0.5	—	6.1
ii)	Residue on sieve, percent by mass, *Max*	0.1	—	7
iii)	Oil absorption	Not less than 11 and not more than 16	—	8
iv)	Colour	Close match to the approved sample	—	9.1
v)	Reducing power	Not inferior to the approved sample	—	11
vi)	Matter soluble in water, percent by mass, *Max*	0.25	—	12
vii)	Sulphur content, percent by mass, *Max*	0.05	B	—

*Methods of test for dry pigments and extenders for paints (*revised).*

*Methods of test for dry pigments and extenders for paints (*revised).*
†Glossary of terms relating to paints (*revised*).

4. PACKING AND MARKING

4.1 Packing — Unless otherwise agreed to between the purchaser and the supplier, the material shall be suitably packed in paper bags.

4.2 Marking — The containers shall be marked with the following information:

a) Name of the material;
b) Manufacturer's name or his recognised trade-mark, if any;
c) Mass of the material;
d) Batch or lot number in code or otherwise;
e) Month and year of manufacture; and
f) Statement if the material is lead free.

4.2.1 The packages may also be marked with the Standard Mark.

4.2.2 The use of the Standard Mark is governed by the provisions of Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5. SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed under **3** of IS : 33-1963*.

6. TEST METHODS

6.1 Tests shall be conducted as prescribed in IS : 33-1963* and in Appendices A and B. Reference to the relevant clauses of IS : 33-1963* is given in col 5 of Table 1.

6.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960†) shall be employed in tests.

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

7. CRITERIA FOR CONFORMITY

7.1 A lot shall be declared as conforming to this standard if the test results of the composite sample satisfy the requirements prescribed under **3.**

*Methods of test for dry pigments and extenders for paints (*revised).*
†Specification for water, distilled quality (*revised).*

APPENDIX A

(*Clause* 3.2)

DETERMINATION OF ZINC OXIDE

A-0. GENERAL

A-0.1 The percentage of zinc oxide in the pigment shall be determined by either volumetric or EDTA method. The former method shall be used as a referee method and the latter for routine analysis.

A-1. VOLUMETRIC METHOD

A-1.1 Outline of the Method — A solution of the material in hydrochloric acid is titrated against standard potassium cyanoferrate (II)* solution and assay calculated.

A-1.2 Reagents

A-1.2.1 *Aqueous Ammonia* — r. d. 0.9 and 4 N.

A-1.2.2 *Hydrochloric Acid*— r. d. 119 and 4 N.

A-1.2.3 *Hydrogen Peroxide Solution* — 3 percent.

A-1.2.4 *Hydtogen Sulphide* — saturated aqueous solution.

A-1.2.5 *Potassium Hexacyanoferrate (II)* Standard Solution* — Approximately 0.05 M. Dissolve 21.0 g of potassium hexacyanoferrate (II), 300 mg of potassium hexacyanoferrate (III)† and 2 R of anhydrous sodium carbonate (to stabilize the solution) in water, and dilute with water to 1 000 ml.

A-1.2.5.1 *Standardization of the potassium hexacyanoferrate (II) solution* — Pipette 25.0 ml of zinc chloride solurion into a flask and add 4 N ammonia until a piece of Congo paper placed in contact with the solution just turns to a pure red colour. Then carefully neutralize the solution by adding hydrochloric acid 4 N from a dropping bottle and add a few drops in excess until the Congo paper turns to a permanent bluish-red or reddish-blue colour *(p*H 3.0 to 1.5). Make up to 150 ml with water, heat the solution to boiling, and add 10 drops of diphenylamine solution. Immediately titrate the solution with potassium cyanoferrate solution until the colour turns to a permanent yellow or yellowish-green (V_1 ml being used). Then back titrate the solution with zinc chloride solution until the colour just turns to blue again *(*V_2 ml being used).

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*International Union of Pure and Applied Chemistry (IUPAC) name for potassium ferrocyanide, K_4 Fe $(CN)_4$.

†International Union of Pure and Applied Chemistry (IUPAC) name for potassium ferricyanide, K_3 Fe $(CN)_4$.

The standardization factor F of potassium cyanoferrate (II) solution expressed in grams of zinc per ml is given by the following formula:

$$F = \frac{0{\cdot}005\ (25 + V_2)^*}{V_1}$$

A-1.2.6 *Zinc Chloride-Standard Solution* — Weigh accurately 5.0 g of chemically pure zinc, dissolve in 300 ml of 4 N hydrochloric acid and dilute with water to 1 000 ml in a volumetric flask.

A-1.2.7 *Diphenylamine Solution in Ethanol* — 5 g per 100 ml.

A-1.2.8 *Congo Paper*

A-1.2.9 *Lead Acetate Paper*

A-1.3 Procedure

A-1.3.1 Accurately weigh about 1.0 g of zinc oxide dried at 105 ± 2°C. Mix with 15 ml of concentrated hydrochloric acid and 30 ml of water and evaporate to dryness. Dissolve the residue, by gentle heating if necessary, in 7 ml of concentrated hydrochloric acid and 30 ml of water. Then add 75 ml of saturated hydrogen sulphide solution, heat the suspension to a temperature of 40°C and allow to stand for one hour at this temperature.

A-1.3.2 When the lead sulphide has settled, filter the liquid into a 500-ml volumetric flask and wash the filter thoroughly with a mixture of 25 ml of the saturated hydrogen sulphide solution, 5 ml of concentrated hydrochloric acid and 75 ml of water. Boil the filtrate and washings to expel hydrogen sulphide (test on lead acetate paper). Cool† the solution, make up to 500 ml with water and shake. Pipette 100 ml of this solution into a flask and add 4 N aqueous ammonia until a piece of Congo paper, added to the solution, just turns to a pure red colour. Carefully add 4 N hydrochloric acid from a dropping bottle to neutralize the solution and add a few drops in excess until the Congo paper turns to a bluish-red or reddish-blue colour (pH 3.0 to 1.5). Heat the solution to boiling, add 10 g of diphenylamine solution and titrate at once, in a similar manner as described in **A-1.2.5.1** [V_3 ml of potassium cyanoferrate (II) solution and V_4 ml of zinc chloride solution being required].

*If the Zinc content of solution in **A-1.2.6** is not exactly 0 005 g/ml an appropriate correction should be applied to the factor 0.005.

†If the solution contains iron or manganese, add, after cooling, about 1 ml of 3 percent hydrogen peroxide and 60 ml of concencrated aqueous ammonia. Make up to 500 ml with water and allow the solution to stand for two hours. Then filter through an absolutely dry filter and funnel. Discard the first 10 to 20 ml of the filtrate. Collect the remainder in a dry flask, remove 100 ml by means of a pipette and boil this to expel ammonia. Then acidify this solution with hydrochloric acid 4 N until a piece of Congo paper turns to a bluish-red or a reddish-blue (*p*H 3.0 to 1.5). Heat the solution to boiling, add 10 drops of diphenylamine solution and immediately titrate the solution in a similar manner as described in **A-1.2.5.1** (V_3 ml of potassium cyanoferrate solution and V_4 ml of zinc chloride solution being used). For calculation, *see* under **A-1.4.**

A-1.4 Calculation

$$\text{Zinc oxide, percent by mass} = 6.223\,(FV_3 - 0{\cdot}005\ V_4)\frac{100^*}{m}$$

where

F = standardization factor of potassium cyanoferrate (II) solution,

V_3 = volume in ml of potassium cyanoferrate (II) solution used,

V_4 = volume in ml of standard zinc chloride solution used,

m = mass in g of the test sample.

A-2. EDTA METHOD

A-2.1 Reagents

A-2.1.1 *Standard Ethylene Diamine Tetraacetate (EDTA) Solution* — 0.015 M. Weigh accurately 5.5 g of disodium ethylene diamine tetraacetate dihydrate, dissolve in water and make up the volume to one litre.

A-2.1.2 *Standard Zinc-Chloride Solution* — 0.015 M. Weigh accurately about 0.9 to 1.1 g of metallic zinc, dissolve in concentrated hydrochloric acid and make up the volume to 1 litre with water.

A-2.1.3 *Eriochrome Black T Indicator* — Dissolve 0.1 g of eriochrome black T in 25 ml of methyl alcohol.

A-2.2 Procedure — Weigh accurately about 0.2 to 0.25 g of zinc oxide sample, dissolve in concentrated hydrochloric acid and make up the volume to 250 ml with water. Pipette out 25 ml of the solution into a 250-ml conical flask and add 50 ml of EDTA solution. Then add 10 ml of ammonia so as to get *p*H 10 to 11. Heat the content to 50 to 60°C. Then titrate against standard zinc chloride solution using eriochrome black T as an indicator. At end-point blue coloured solution changes to light pink coloured solution. Carry out a blank using only 50 ml of EDTA solution under similar conditions.

A-2.3 Calculation

$$\text{Zinc oxide, percent by mass} = \frac{(B - V) \times m \times 81{\cdot}38}{M \times 65{\cdot}38}$$

where

B = volume in ml of standard zinc chloride solution used in the blank titration,

*If the zinc content of solution in **A-1.2.6** is not exactly 0.005 g/ml an appropriate correction should be applied to the factor 0.005.

V = volume in ml of standard zinc chloride solution used in titration using the sample,
m = mass in g of metallic zinc per litre used in preparation of zinc chloride solution, and
M = mass in g of sample taken for the test.

APPENDIX B

[*Table* 1, *Item* (vii)]

DETERMINATION OF SULPHUR CONTENT

B-0. GENERAL

B-0.1 Outline of the Method — A known quantity of the material is treated with bromine water in presence of hydrochloric acid, boiled and again heated in presence of granular aluminium, filtered and washed. The filtrate is neutralized with ammonium hydroxide, reacidified and boiled. Sulphur is precipitated as barium sulphate by adding barium chloride solution, filtered, dried and sulphur content is calculated.

B-1. REAGENTS

B-1.1 Bromine Water — saturated.

B-1.2 Aluminium — granular aluminium, chemically pure.

B-1.3 Barium Chloride Solution — 100 g of barium chloride per litre. Dissolve 117 g of barium chloride crystals ($BaCl_2$. $2H_2O$) in water and dilute to 1 litre.

B-2. PROCEDURE

B-2.1 Transfer about 10 g of the sample, weighed to the nearest 0.1 g to a 400-ml beaker. Add 50 ml of saturated bromine water, 100 ml of water, and 25 ml of hydrochloric acid. Boil until all the bromine has been expelled, cool, and add 3 to 5 g of granular aluminium. Heat to boiling, filter, and wash well with hot water.

B-2.2 Dilute the filtrate to 300 ml with water, neutralize with ammonium hydroxide solution and add 6 drops of hydrochloric acid. Heat to boiling and add 25 ml of hot barium chloride solution dropwise, with constant stirring. Allow to stand in a warm place for at least 2 hours.

B-2.3 Filter, using a weighed Gooch crucible, and wash well with hot water. Dry and ignite in a muffle furnace for 30 minutes. Cool and weigh as barium sulphate.

B-3. CALCULATION

$$\text{Sulphur content, percent by mass} = \frac{M_1 \times 0{\cdot}1374}{M_2} \times 100$$

where

M_1 = mass in g of barium sulphate, and
M_2 = mass in g of sample taken.

(*Continued from page* 2)

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