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Draft Indian Standard

ZINC CHROME PIGMENTS FOR PAINTS — SPECIFICATION

(Fifth Revision of IS 51)

(ICS 87.060.10)

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

Last Date for Comments: 18th April 2024

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FOREWORD

(Formal Clause shall be added later)

This standard was published in 1950 and subsequently revised in 1963, 1966 and 1979. In the fourth revision in 1998, Type 1 zinc chrome was deleted, not being in vogue and the other types redesignated.

This fifth revision has been undertaken to align the product standards with current market demands, enhancing its acceptance and relevance. Notable changes include:

- The scope of the standard has been refined to precisely encompass the material's intended use;
- The oil absorption requirement has been revised from a maximum of 15 percent to within ± 10 percent of the approved sample. Additionally, the Alkali metal content requirement has been revised to a maximum of 12 percent, replacing the previous range of 10.0 percent to 12.0 percent;
- The test method for colour and strength measurement has been changed to Vibro Shaker method in place of existing test method based on Muller. Additionally, in colour tinting strength, staining power and tone and ease of dispersion have been incorporated.
- To address health and safety concerns, restrictions for lead has been tightened and restriction for toxic heavy metals has been introduced;
- 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.

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 CHROME PIGMENT FOR PAINTS — SPECIFICATION***(Fifth Revision)***1 SCOPE**

This standard prescribes requirements and methods of sampling and test for zinc chromate pigments for paints and/or corrosion-inhibiting coatings.

2 REFERENCES

The Indian Standards listed in Annex A contain provisions which through reference in this text, constitute provisions of this Indian 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 indicated in Annex A.

3 TERMINOLOGY

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

4 TYPES

4.1 The material shall be of the following two types:

- a) *Type 1* — Basic potassium zinc chromate of approximate composition K_2CrO_4 , $3ZnCrO_4$, $Zn(OH)_2$, intended for use mainly as anti-corrosive pigment for its rust-inhibiting properties in general (colour Index Pigment Yellow 36).
- b) *Type 2* — Basic zinc chromate free from alkali metals of approximate composition $ZnCrO_4$, $4Zn(OH)_2$, intended for use mainly as anti-corrosive pigment for its rust-inhibiting properties generally in etch primers (colour Index Pigment Yellow 36.1).

5 REQUIREMENTS**5.1 Form and Condition**

The material shall be in the form of dry powder or in such condition that it is possible to reduce it to powder form by crushing, without grinding action, under a palette knife.

5.2 Composition**5.2.1 Type 1**

The material shall consist essentially of basic potassium zinc chromate and shall be of approximate composition K_2CrO_4 , $3ZnCrO_4$, $Zn(OH)_2$.

5.2.2 Type 2

The material shall consist essentially of basic zinc chromate free from alkali metals and shall be of approximate composition $ZnCrO_4$, $4Zn(OH)_2$.

5.2.3 The material shall be free from organic dyestuff or substitute of any kind and shall be completely soluble in 10 percent acetic acid at a temperature of 80°C.

5.3 Lead Restriction

The material shall be tested for restriction from lead in accordance with ICP-OES or AAS method of IS 101 (Part 8/Sec 5). When thus tested the material shall not contain lead or compounds of lead or mixtures of both, calculated as metallic lead exceeding 90 ppm.

NOTE — When no lead is used during production, the product is considered lead free.

5.4 Toxic Heavy Metal Restriction

Product shall not be manufactured using mercury and mercury compounds, cadmium, chromium VI, arsenic, antimony, and their oxides. The material shall not contain more than 0.1 percent by weight in total of above toxic heavy metals in the form of natural impurities or impurities entailed by the production process which are contained in the raw material when tested by the relevant Atomic Absorption Spectroscopic methods.

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

Table 1 Requirement for Zinc Chrome

(Clauses 5.5, 8.1 and B-3.3)

Sl No.	Characteristic	Requirement		Method of Test, Ref to
		Type 1	Type 2	
(1)	(2)	(3)	(4)	(5)
i)	Volatile matter, percent by mass, <i>Max</i>	1.0	1.0	IS 33
ii)	Residue on sieve, percent by mass, <i>Max</i>	0.5	0.5	IS 33
iii)	Oil absorption, <i>Max</i>	±10 percent of approved sample		IS 33
iv)	Matter insoluble in ammoniacal ammonium chloride solution, percent by mass, <i>Max</i>	0.5	0.5	B-2 of Annex B
v)	Combined water, percent by mass, <i>Max</i>	8.0	—	B-3 of Annex B
vi)	Chromic anhydride (CrO ₃), percent by mass, <i>Max</i>	43.0	17	B-4 of Annex B
vii)	Total zinc content (as ZnO), percent by mass	36.5 to 40	68.5 to 72.0	B-4 of Annex B
viii)	Alkali metal content (as K ₂ O), percent by mass, <i>Max</i>	12	—	B-5 of Annex B
ix)	Water soluble sulphates (as SO ₄), percent by mass, <i>Max</i>	0.1	0.1	B-6 of Annex B
x)	Water soluble chlorides (as Cl), percent by mass, <i>Max</i>	0.1	0.1	B-6 of Annex B
xi)	Water soluble nitrates (as NO ₃), percent by mass, <i>Max</i>	0.1	0.1	B-6 of Annex B

xii)	Water soluble chromates (as CrO_3), percent by mass	0.25 to 0.50	—	B-7 of Annex B
xiii)	Colour a) Tinting strength b) Staining power and tone c) Ease of dispersion	Agreed between purchaser and supplier		B-8 of Annex B
xiv)	Matter soluble in water, percent by mass, <i>Max</i>	—	0.5	IS 33

6 PACKING AND MARKING

6.1 Packing

The material shall be suitably packed as agreed to between the purchaser and the supplier.

6.2 Marking

6.2.1 The containers shall be marked with the following information:

- a) Name of the material;
- b) Type of the material;
- c) Net mass of the material;
- d) Batch No. or Lot No. in code or otherwise; and
- e) Month and year of manufacture
- f) Expiry date/shelf life/best before
- g) Lead content (Maximum);
- h) Toxic heavy metals content
- i) A cautionary note as below:
 - a. Keep out of reach of children; or
 - b. This product may be harmful if swallowed or inhaled

6.2.2 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 there under, and the products may be marked with the Standard Mark.

7 SAMPLING

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

7.2 Criteria for Conformity

The material shall be taken as conforming to this specification if the composite sample satisfies all the requirements prescribed.

8 TEST METHODS

8.1 Tests shall be conducted as prescribed in IS 33 and in Annex B, according to the requirements of Table 1, **5.3** and **5.4**.

8.2 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

(Clause 2)

LIST OF REFERRED STANDARD

IS No.	Title
IS 33 : 1992	Inorganic pigments and extenders for paints — Methods of sampling and test (<i>third revision</i>)
IS 101 (Part 4/Sec 2) : 2021	Methods of sampling and test for paints varnishes and related products: Part 4 optical tests, Sec 2 Colour-visual comparison of colour of paints (<i>fourth revision</i>)
IS 264 : 2005	Nitric acid — Specification (<i>third revision</i>)
IS 266 : 1993	Sulphuric acid — Specification (<i>third revision</i>)
IS 323 : 2009	Rectified spirit for industrial use — Specification (<i>second revision</i>)
IS 1070 : 2023	Reagent grade water — Specification (<i>fourth revision</i>)
IS 1303 : 1983	Glossary of terms relating to paints (<i>second revision</i>)
IS 2316 : 1990	Methods of preparation of standard solutions for calorimetric and volumetric analysis (<i>second revision</i>)

ANNEX B

(Table 1 and Clause 8.1)

ANALYSIS OF ZINC CHROME FOR PAINTS

B-1 PREPARATION OF SAMPLE

Grind the pigment, if it is lumpy or not finely ground, to a fine powder. If the sample is large, mix thoroughly and take a representative portion.

B-2 DETERMINATION OF MATTER INSOLUBLE IN AMMONIACAL AMMONIUM CHLORIDE SOLUTION

B-2.1 Outline of the Method

Matter insoluble in ammoniacal ammonium chloride solution is determined by drying the residue obtained after shaking the material with ammoniacal ammonium chloride solution.

B-2.2 Apparatus

B-2.2.1 Sintered Glass Crucible (G No. 4) or Gooch Crucible**B-2.3 Reagent****B-2.3.1 Ammoniacal Ammonium Chloride Solution**

Dissolve 30 g of ammonium chloride in a mixture of 500 ml of ammonium hydroxide of relative density 0.92 and 600 ml of water.

B-2.4 Procedure

Shake approximately 5 g of the sample, accurately weighed, with 100 ml of the reagent for 1 h at room temperature. Allow to stand for 30 min and decant the supernatant liquid through a tared sintered glass or Gooch crucible. To the residue add a further 100 ml of reagent, shake for 5 min and filter the whole suspension through the same crucible, any adherent residue being transferred to the crucible by means of a policeman and with the minimum additional reagent. Wash the sides of the crucible and the residue with two successive portions of 25 ml water, and then dry the crucible and the residue to constant mass in an oven maintained at $(100 \pm 2) ^\circ\text{C}$.

B-2.5 Calculation

Matter insoluble in ammoniacal ammonium chloride solution,

$$\text{percent by mass} = \frac{100 m}{M}$$

where

m = mass in g of residue, and

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

B-3 DETERMINATION OF COMBINED WATER**B-3.1 Outline of the Method**

Combined water is determined by direct combustion of material at $1\,000\,^\circ\text{C}$ and making correction for the volatile matter.

B-3.2 Procedure

Place about 2 g of the sample, accurately weighed, in a porcelain boat. Introduce the boat into a combustion tube of an electrically heated combustion furnace of the type used for the determination of carbon by direct combustion. Connect the combustion furnace to a source of pure dry air at one end and an accurately weighed train of absorption tubes containing dry fused calcium chloride free from lime at the other end. Heat the combustion tube to $1\,000\,^\circ\text{C}$ and continuously pass a current of dry air. After heating for 1 h, cool the tube. Reweigh the absorption train. Repeat this operation till constant mass of the absorption train is obtained. The increase in mass gives the combined water and volatile matter present in the material.

B-3.3 Calculation

Combined water,

$$\text{percent by mass} = \frac{m}{M} \times (100 - V)$$

where

m = increase of mass in g of the absorption train,

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

V = percent by mass of volatile matter (*see* Table 1).

B-4 DETERMINATION OF CHROMIC ANHYDRIDE AND ZINC OXIDE CONTENT

B-4.1 Outline of the Method

Chromic anhydride is determined by titrating the iodine liberated from potassium iodide with standard sodium thiosulphate solution. Zinc oxide is determined in the same solution by titrating the iodine subsequently liberated on addition of potassium ferricyanide with standard sodium thiosulphate solution.

B-4.2 Reagents

B-4.2.1 *Dilute Sulphuric Acid* — Approximately 7 N.

B-4.2.2 *Potassium Iodine Solution (Freshly prepared)* — 10 percent.

B-4.2.3 *Standard Sodium Thiosulphate Solution* — 0.1 N.

B-4.2.4 *Starch Solution* — 0.5 percent.

B-4.2.5 *Ammonia Solution* — 1 : 1 (v/v).

B-4.2.6 *Potassium Ferricyanide Solution M/10* – Prepared by dissolving 8.231 g of potassium ferricyanide in 250 ml cold distilled water, filtered rapidly and kept in a dark bottle. The solution can be suitably used for 7 days if kept in dark when not in use.

B-4.2.7 *Ammonium Bifluoride* (NH_4HF_2) — Solid.

B-4.3 Procedure

B-4.3.1 Weigh accurately about 0.5 g of the material in a 500 ml conical flask. Add 70 ml of water and 30 ml of dilute sulphuric acid. Boil slowly for 2 min to 3 min and cool.

B-4.3.2 Transfer the solution to a 500 ml iodine flask containing 100 ml of water and 20 ml dilute sulphuric acid. Add 30 ml of potassium iodide solution and allow to stand for 5 min. Titrate the liberated iodine with standard sodium thiosulphate solution after adding 5 ml of starch solution as indicator. Note the volume of sodium thiosulphate solution used in the titration (V_1).

B-4.3.3 After the titration for chromic anhydride, add ammonia solution dropwise until the litmus test paper first turns blue. (An excess of ammonia solution, more than 3 drops to 4 drops, should not be added as it is essential that the blue litmus should turn red with ammonium bifluoride to be added subsequently.) Cool and add 2 g to 3 g of ammonium bifluoride and sufficient water to make up the volume to approximately 250 ml. (Sometime, traces of iodine liberated during neutralization can be cleared by adding one or two drops of sodium thiosulphate solution.) Add $V_1/3$ ml of potassium ferricyanide solution. Stir and allow to stand for about 2 min. Titrate the liberated iodine with standard sodium thiosulphate solution. After the end point the colour of the test solution will be greenish yellow. Note the volume of sodium thiosulphate solution used in this second titration (V_2).

B-4.3.3.1 In case V_2 is less than 6 ml, the titration is to be repeated with the addition of $V_2 + 2$ ml of potassium ferricyanide solution in place of $V_1/3$ ml.

B-4.3.3.2 In case V_2 is less than 3 ml, the test is to be repeated, starting from the sample, with the addition of 5 ml potassium ferricyanide solution.

B-4.4 Calculation

Chromic anhydride (CrO_3),

$$\text{percent by mass} = \frac{3.334 \times V_1 \times N}{M}$$

Zinc content (as ZnO),

$$\text{percent by mass} = \frac{12.45 \times V_2 \times N}{M}$$

where

V_1 = volume in ml of sodium thiosulphate solution used in first titration,

V_2 = volume in ml of sodium thiosulphate solution used in second titration,

N = normality of sodium thiosulphate solution used, and

M = mass in g of the material taken for titration.

B-5 DETERMINATION OF ALKALI METALS**B-5.1 Outline of the Method**

Alkali metals are determined volumetrically by titrating the excess of standard ferrous ammonium sulphate in the alkali chromate solution, obtained by heating the material at high temperature, against standard potassium permanganate solution.

B-5.2 Reagents**B-5.2.1 Dilute sulphuric Acid**

Dilute 25 ml of sulphuric acid (*see* IS 266) by adding to water, cooling and making up to 100 ml with water.

B-5.1.2 Standard Ferrous Ammonium Sulphate Solution,

0.1 N (*see* IS 2316).

B-5.2.3 Standard Potassium Permanganate Solution,

0.1 N (*see* IS 2316).

B-5.3 Procedure

Heat 2.0 g of the material to approximately 600 °C for 1 h. The material is thereby decomposed into zinc oxide, chromium sesquioxide (Cr_2O_3) and alkali metal chromate. Cool and extract the alkali metal chromite from the residue with hot water, filter and wash free from soluble chromate. Add a measured excess volume (usually 100 ml is sufficient) of ferrous ammonium sulphate solution and titrate the excess with standard potassium permanganate solution.

B-5.4 Calculation

Alkali metal (as K_2O),

$$\text{percent by mass} = 0.1565 (V_1 - V_2)$$

where

V_1 is volume in ml of standard ferrous ammonium sulphate solution added, and

V_2 is volume in ml of standard potassium permanganate solution required for titration.

B-6 DETERMINATION OF WATER SOLUBLE SULPHATES, CHLORIDES AND NITRATES

B-6.1 Outline of the Method

Water-soluble sulphates and chlorides are determined gravimetrically as barium sulphate and silver chloride respectively. Water-soluble nitrates are determined calorimetrically with Nessler's reagent.

B-6.2 Apparatus

B-6.2.1 *Distillation Flask* — 500 ml capacity.

B-6.2.2 *Mechanical Shaker*

B-6.2.3 *Nessler Tubes* — 50 ml capacity.

B-6.3 Reagents

B-6.3.1 *Hydrochloric Acid* — Approximately 5 N.

B-6.3.2 *Ethyl Alcohol* — 95 percent (v/v) or rectified spirit (*see* IS 323).

B-6.3.3 *Barium Chloride Solution* — 10 percent, obtained by dissolving 12.0 g barium chloride crystals in water and making up to 100 ml.

B-6.3.4 *Concentrated Nitric Acid* — (*see* IS 264).

B-6.3.5 *Silver Nitrate Solution* — Approximately 5 percent (m/v).

B-6.3.6 *Devarda's Alloy* — Containing 45 parts of aluminium, 50 parts of copper and 5 parts of zinc. Heat the aluminium in a crucible in a furnace until it begins to melt. Add copper in small portions and heat until liquefied. Now plunge zinc into the molten mass. Cover the crucible, heat the mixture for a few minutes and stir with an iron rod. Allow it to cool slowly with the cover on and then pulverize the crystalized mass.

B-6.3.7 *Sodium Hydroxide Solution* — Approximately 20 percent (m/v).

B-6.3.8 *Nessler's Reagent* — Dissolve 10 g of potassium iodide in 10 ml of water and add to it slowly, with stirring, a saturated aqueous solution of mercuric chloride (HgCl_2) until a slight permanent precipitate forms. Add 30 g of potassium hydroxide, and when it has dissolved, add 1 ml more of mercuric chloride and then dilute to 200 ml with water. Allow to settle overnight and then decant the clear solution. Keep the solution in a bottle closed with well-fitting rubber stopper.

B-6.3.9 *Standard Ammonium Chloride Solution* — Take 0.017 g of ammonium chloride, dissolve in water and dilute in a graduated flask to 1 000 ml.

B-6.4 Procedure

B-6.4.1 *Preparation of Solution*

Shake 20 g of the material with 200 ml of water in a chemically resistant glass flask for 1 hour on a mechanical shaker. Filter the mixture and *serve the filtrate for determinations given in **B-6.4.2** to **B-6.4.4**.

B-6.4.2 *Determination of Sulphates*

Acidify 50 ml of the clear aqueous extract with 3 ml of hydrochloric acid and add a few millilitres of ethyl alcohol. Warm the solution to reduce the chromate which is noted by a change in colour to green. Boil the solution vigorously to drive off organic compounds, care being taken that the solution does not splash. Add excess of barium chloride solution and allow to stand overnight. Filter the precipitated barium sulphate through a Gooch crucible. Wash the precipitate with hot water until washings are free of chloride ions. Dry the crucible containing the precipitate and ignite it over a burner or in the muffle furnace at 600 °C to 700 °C for 30 min. Cool in a desiccator and weigh.

B-6.4.2.1 Calculation

Water-soluble sulphate (as SO₄)

$$\text{percent by mass} = 8.23 M$$

where M is mass in g of barium sulphate precipitate.

B-6.4.3 Determination of Chlorides

Acidify 50 ml of the clear aqueous extract obtained as in **B-6.4.1** with 10 ml of concentrated nitric acid and add a slight excess of silver nitrate solution. Coagulate the precipitated silver chloride by warming. Protect the precipitate from light by wrapping with black paper. Filter the precipitate through a sintered glass or Gooch crucible. Wash the precipitate with water containing traces of nitric acid and dry at (100 ± 5) °C to constant mass.

B-6.4.3.1 Calculation

Water-soluble chlorides (as Cl),

$$\text{percent by mass} = 4.96 M$$

where M is mass in g of silver chloride precipitate.

B-6.4.4 Determination of Nitrates

Place 50 ml of the clear aqueous extract obtained as in **B-6.4.1** in a distillation flask and dilute to 150 ml with water. Add 3.0 g of powdered Devarda's alloy and 30 ml of sodium hydroxide solution and close the apparatus at once. Warm the flask gently until the reaction starts and allow the reaction to proceed gently for about 30 min. Distil the mixture, collecting the distillate in a receiver containing 30 ml of water and 2 ml of concentrated hydrochloric acid, until about 70 ml of water has distilled over. Keep the receiver cool with running water. Dilute the distillate to 250 ml. Transfer 5 ml of this solution to a Nessler tube and to this add 1 ml of Nessler's reagent. Match the colour produced with a series of similar Nessler tubes to which varying measured quantities of standard ammonium chloride solution and 1 ml of Nessler's reagent have been added.

B-6.4.4.1 Calculation

$$\text{Water-soluble (as NO}_3\text{), Percent by mass} = 0.02 V$$

where V is volume in ml of standard ammonium chloride solution required to match the colour.

B-7 DETERMINATION OF WATERSOLUBLE CHROMATES

B-7.1 Outline of the Method

Water-soluble chromates are determined volumetrically by adding an excess of ferrous ammonium sulphate to the water extract of the material and then titrating the excess against standard potassium permanganate solution.

B-7.2 Apparatus

B-7.2.1 Mechanical Shaker

B-7.3 Reagents

B-7.3.1 Dilute Sulphuric Acid

Dilute 25 ml of sulphuric acid (*see* IS 266) by adding to water, with cooling, and making up to 100 ml with water.

B-7.3.2 Standard Ferrous Ammonium Sulphate Solution

0.1 N (*see* IS 23 16).

B-7.3.3 Standard Potassium Permanganate Solution

0.1 N (*see* IS 23 16).

B-7.4 Procedure

Shake 30 g of the sample with 100 ml of water for 1 h on a mechanical shaker at $(27 \pm 2) ^\circ\text{C}$. Filter and transfer 50 ml of the clear filtrate to a conical flask and acidify with sulphuric acid. Add a measured excess volume (usually 50 ml is sufficient) of the standard ferrous ammonium sulphate solution and titrate the excess against standard potassium permanganate solution.

B-7.5 Calculation

Water-soluble chromates (as CrO_3),

$$\text{percent by mass} = 0.02223 (V_1 - V_2)$$

where

V_1 = volume in ml of standard ferrous ammonium sulphate solution added, and

V_2 = volume in ml of standard potassium permanganate solution required for titration.

B-8 DETERMINATION OF COLOUR, STRENGTH AND DEGREE OF DISPERSION/FINENESS OF GRINDING

B-8.1 Outline of the Method

To evaluate the performance of pigment with respect to colour difference, strength, and degree of dispersion against approved pigment sample

B-8.2 Apparatus

B-8.2.1 Vibroshaker — 25 degree to 30 degree angular oscillation @ 640 / 660 spm simultaneously

B-8.2.2 Closed SS metal container — 125 ml

B-8.2.3 Glass Beads — diameter 2.5 mm to 3.0 mm

B-8.2.4 Electronic Balance — accuracy 1 mg

B-8.2.5 Spatula**B-8.2.6 Mechanical Stirrer****B-8.2.7 Black and White Paper****B-8.2.8 Bar Coater K-7****B-8.3 Procedure****B-8.3.1 Method of Preparation of the Pigment Concentrate:**

- Add the material in 0.4 factor and add 60 g of glass beads^e in a closed SS metal container 125 ml
- Dispersion time after addition of Stage 1: 40 min.
- After dispersion, add Stage 2 and run for another 10 min.
- Collect the pigment concentrate and proceed for following testing:

Table – 2 Guideline Formulation of Preparation of Pigment Concentrate

Sl. No	Component	Weight (%)	Mixing Time
i)	Pigment	20.0 ± 0.001	
Stage 1			
ii)	Epoxy Resin [*]	8.0	Add in sequence and disperse for 40 minutes
iii)	n-Butanol	4.0	
iv)	2-Ethoxy ethanol	1.0	
v)	Aromatic Hydrocarbon Solvent C9	6.0	
vi)	Rheological additive for moderate to high polarity organic Media [#]	0.8	
vii)	Wetting and dispersing additive ^{\$}	1.0	
Stage 2			
viii)	Epoxy Resin [*]	28.0	Add in sequence and disperse for 10 minutes
ix)	n-Butanol	6.0	
x)	2-Ethoxy ethanol	1.0	
xi)	Aromatic Hydrocarbon Solvent C9	24.2	
Total		100.0	
[*] High-viscosity unmodified epoxy resin of bisphenol-A type at 75 percent solids in xylene, Viscosity of solution at 25°C 8.0 Pa.s to 13.0 Pa.s and Epoxy equivalent 450 g/eq to 525 g/eq.			
[#] Organic derivative of a bentonite clay			
^{\$} Alkyl ammonium salt of a high molecular weight copolymer (Amine value: (44 ± 5) mg KOH/g, Acid value: (38 ± 5) mg KOH/g)			
^e Use glass beads as grinding media 60 g			

B-8.4 Determination of Degree of Dispersion/Fineness of Grinding:

To assess the degree of dispersion/fineness of grinding by Hegman Gauge:

Fineness of grinding is expressed as maximum crowing of particles on a Hegman Scale of 0 to 8.

- a) Ensure that the Gauge, Scraper, and Spatula are clean.
- b) Place the Hegman Gauge on a flat surface.
- c) Put the sample under test into the deep end of channel with spatula.
- d) Hold the scraper between the thumbs and fingers of both hands.
- e) Ensure that the scraper is perpendicular to the surface of the gauge and place it edgewise in contact with the surface.
- f) Draw the paint at a uniform rate down the gauge passing the mark 8, within 2 s to 3 s.
- g) View the gauge sideways by reflected light as quickly as possible (withing 10 s) and read the reading.
- h) Hegman Gauge reading is defined as the point where crowding particles may be seen in a line across the gauge.

B-8.5 Determination of Colour and Strength %

B-8.5.1 Colour Difference (Mass Tone)

B-8.5.1.1 To check the colour difference of pigment concentrate it need to be cured with catalyst. Catalyst part contains some reactive group that reacts with the resin (binder) to build high molecular weight and initiate curing reaction.

B-8.5.1.2 Catalyst — Medium viscosity polyamide hardener

B-8.5.1.3 Amine Value — (300 ± 20) mg KOH/g

B-8.5.2 Calculation of Mixing Ratio:

Epoxy equivalent of Epoxy resin = E

Solid content of Epoxy resin = X%

Loading of Epoxy resin in formulation (Table – 1) = Y%

$$\text{Active content} = \frac{Y \times X}{100}$$

Amine value of catalyst: C

$$\text{Amine equivalent weight} = \frac{56100}{C}$$

$$\text{E.g Epoxy cured with } \frac{56100}{C} \text{ g Amine}$$

$$1 \text{ g Epoxy cured with } \frac{56100}{C \times E} \text{ g Amine}$$

$$\frac{Y \times X}{100} \text{ Epoxy cured with } \frac{56100 \times Y \times X}{100 \times C \times E} \text{ g Amine}$$

$$\text{Therefore, 100 g Pigment concentrate cured with } \frac{561 \times Y \times X}{C \times E} \text{ g Amine}$$

Mixing Ratio of Pigment concentrate:

$$\text{Catalyst (w/w)} = 100 : \frac{561 \times Y \times X}{C \times E}$$

B-8.5.3 Procedure

- Mix the pigment concentrate and catalyst as per above mentioned ratio and mix uniformly with a spatula by hand mixing with minimum foam generation.
- Make drawdowns side by side (approved sample vs. sample under evaluation) with the help of K-7 bar coater.
- Conduct a rub out test by finger on wet film at both upper corners of the drawdown paper (approved and sample both) and observe for difference in colour depth/intensity between rub out and adjacent areas to check colour acceptability.
- Dry the drawdown paper at 80 °C oven for 30 min for curing. After drying, check the colour difference (mass tone) of the specimen against approved sample both visually as well as in Spectro (CIE Lab. D65 10⁰ observer).

B-8.6 Colour Difference (Reduced Tone) and Strength%

B-8.6.1

To check the reduced tone and strength% we need an epoxy based white base. The white base can be prepared following the same guideline formulation of preparation of pigment concentrate (Table 3) where pigment should be Rutile.

Reduced tone and Strength% [White Base : Pigment concentrate = 60 : 40 (w/w)]	
White Base (g)	Pigment concentrate (g)
30 ± 0.001	20 ± 0.001

Catalyst: Medium viscosity polyamide hardener

Amine Value: (300 ± 20) mg KOH/g

B-8.6.2 Calculation of Mixing Ratio:

Epoxy equivalent of Epoxy resin = E

Solid content of Epoxy resin = X percent

Loading of Epoxy resin in formulation (Table 3) = Y percent

$$\text{Active content} = \frac{Y \times X}{100}$$

Amine value of catalyst: C

$$\text{Amine equivalent weight} = \frac{56100}{C}$$

E.g Epoxy cured with $\frac{56100}{C}$ g Amine

1 g Epoxy cured with $\frac{56100}{C \times E}$ g Amine

$$\frac{Y \times X}{100} \text{ Epoxy cured with } \frac{56100 \times Y \times X}{100 \times C \times E} \text{ g Amine}$$

Therefore, 100 g Pigmented base cured with $\frac{561 \times Y \times X}{C \times E}$ g Amine

Mixing Ratio of Pigmented base : Catalyst (w/w) = 100 : $\frac{561 \times Y \times X}{C \times E}$

B-8.6.3 Procedure

- a) Mix the pigmented base and catalyst as per above mentioned ratio and mix uniformly with a spatula by hand mixing with minimum foam generation.
- b) Make drawdowns side by side (approved sample vs. sample under evaluation) with the help of K-7 bar coater.
- c) Conduct a rub out test by finger on wet film at both upper corners of the drawdown paper (approved and sample both) and observe for difference in colour depth/intensity between rub out and adjacent areas to check colour acceptability.
- d) Dry the drawdown paper at 80 °C oven for 30 min for curing. After drying, check the colour difference (reduced tone) and strength percent of the specimen against approved sample both visually as well as in Spectro (*see* IS 101 Part 4/ Sec 2).