

BUREAU OF INDIAN STANDARDS

DRAFT FOR COMMENTS ONLY

(Not to be reproduced without permission of BIS or used as an Indian Standard)

Draft Indian Standard

ULTRAMARINE BLUE FOR PAINTS — SPECIFICATION

(Second Revision of IS 55)

(ICS 87.060.10)

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

Last Date for Comments: 26 November 2024

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

FOREWORD

(Formal Clause shall be added later)

This standard was first published in 1950, largely based on the *interim* co-ordinated draft produced with the assistance of representatives of manufacturers and of 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).

Subsequently, first revision was done in 1970. During that revision, a limit for free sulphur was introduced along with its test method. Additionally, an identification test to qualitatively assess the material and tests for fastness to light, soluble organic colouring matter, dispersibility were also added.

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

- a) The material has been classified in the two grades based on free sulphur content.
- b) Requirement for residue on sieve and matter soluble in water have been updated for both the grades.
- c) The test method for color difference, tinting strength, and ease of dispersion has been changed to the Vibro Shaker method, replacing the existing method based on Muller. The requirements have been accordingly modified to reflect this change.

- d) New requirements of density and *pH* value of the aqueous suspension have been added. The requirement of alkalinity has been withdrawn, as it is no longer significant with the introduction of the *pH* requirement.
- e) To address health and safety concerns, restrictions for lead and toxic heavy metals have been introduced;
- f) A suitable precautionary note has been added in the marking clause in order to prevent unforeseen events, and;
- g) Additionally, 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

ULTRAMARINE BLUE FOR PAINTS — SPECIFICATION

(Second Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for the material commercially known as ultramarine blue, intended for use as a paint pigment.

2 REFERENCES

The standards listed in Annex A 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 in Annex A.

3 TERMINOLOGY

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

4 GRADE

The material shall be two grades based on free sulphur content, namely;

- a) **Grade A** — maximum 0.05 percent (mass fraction) free sulphur;
- b) **Grade B** — maximum 0.20 percent (mass fraction) free sulphur.

5 REQUIREMENTS

5.1 Form and Condition

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

5.2 Composition

The material shall be of uniform character and shall consist solely of compounds of sodium, aluminium, silicon, sulphur and oxygen. It is a complex of sodium silicate containing sulphur, and aluminium.

5.3 Identification Test

Warm gently approximately 0.1 g of the material with 1:1 (v/v) hydrochloric acid in a test-tube. The material shall be identified as ultramarine blue, if the colour is destroyed completely with the evolution of hydrogen sulphide gas, detected by its characteristic brown colouration appearing on a strip of moistened lead acetate paper held above the test tube. Any colour remains after this treatment, it may be interpreted that a foreign pigment is present.

5.5 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.

5.6 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 Absorption/Emission Spectroscopic methods.

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

TABLE 1 REQUIREMENTS FOR ULTRAMARINE BLUE FOR PAINTS

(Clause 5.7)

SL No.	CHARACTERISTIC	REQUIREMENT		METHOD OF TEST	
		Grade A	Grade B	Ref to Annex	IS
(1)	(2)	(3)	(4)	(5)	(6)
i)	Volatile matter at (105 ± 2) °C, percent by weight, <i>Max</i>	1.0	1.0	—	33
ii)	Free sulphur content, percent by mass, <i>Max</i>	0.05	0.20	B	—
iii)	Residue on sieve at 63 µm, percent by weight, <i>Max</i>	0.1	0.5	—	33
iv)	Oil absorption	30 to 40†		—	33
v)	Ease of dispersion/ Fineness of grind, microns, <i>Max</i> (by Hegman gauge scale 0 to 8, <i>Min</i>)	15 (6.5)		C	—
vi)	Colour			C	—
	a) Tinting strength, percent	95 – 105 of approved sample/ as agreed between the parties			—
	b) Colour difference (Mass tone), ΔE, <i>Max</i>	1.5			

vii)	Matter soluble in water, percent by weight, <i>Max</i>	1.0	1.5	—	33
viii)	Fastness to light	Not inferior to the approved sample/as agreed between the parties		D	—
ix)	Soluble organic colouring matter	To pass the test		E	—
x)	Density at 23° C, g/cm ³	2.23 to 2.40		—	33
xi)	pH value of aqueous suspension	As agreed between the parties		—	33
†It shall be, however, within ± 10 percent of the approved sample or declared value.					

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) The name of the material;
- b) Manufacturer's name or trade-mark, if any;
- c) Weight of the material; and
- d) The lot and the batch number.
- e) Lead content (Maximum);
- f) Toxic heavy metals content
- g) A cautionary note as below:
 - i) Keep out of reach of children; or
 - ii) This product may be harmful if swallowed or inhaled

6.2.2 The packages may also be marked with the BIS Certification Mark.

6.2.3 *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

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

8 TEST METHODS

8.1 Tests shall be conducted as prescribed in IS 33 and in Annexes B to E mentioned in 5.4 and col 5 and 6 of Table 1.

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 STANDARDS

<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 35 : 2021	Zinc Oxide for Paints — Specification (<i>second revision</i>)
IS 75 : 1973	Specification for linseed oil, raw and refined (<i>second 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 101 (Part 8/Sec 5) : 2022	Methods of sampling and test for paints varnishes and related products : Part 8 tests for pigments and other solids, Sec 5 lead restriction Test (<i>Fourth 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 1997 : 2008 ISO 385	Laboratory glassware - Burettes (<i>third revision</i>)
IS 5296 : 1995	Chloroform, pure and technical - Specification (<i>second revision</i>)

ANNEX B

[Table 1, Item No. (ii)]

DETERMINATION OF FREE SULPHUR

B-1 GENERAL

B-1.1 Outline of Method — The material is extracted with chloroform, as solvent in soxhlet apparatus, and the solvent evaporated from the extracted material. The amount of free sulphur is calculated from the residue left after evaporation.

B-2 APPARATUS

B-2.1 Soxhlet Apparatus

B-3 REAGENT

B-3.1 Chloroform — neutral and pure (*see* IS 5296).

B-4 PROCEDURE

B-4.1 Weigh accurately about 60 g of the material and extract with sufficient quantity of chloroform in a soxhlet apparatus for about 4 hours. Distil off the contents and evaporate the residue to dryness at 60 °C. Weigh the residue obtained.

B-4.2 Calculation

$$\text{Free sulphur, percent by weight} = 100 \times \frac{W_1}{W_2}$$

where

W_1 = weight in g of residue, and

W_2 = weight in g of the material taken.

ANNEX C

[*Table 1, Item (v), (vi)*]

DETERMINATION OF COLOUR, TINTING STRENGTH, COLOUR DIFFERENCE (MASS TONE) AND EASE OF DISPERSION

C-1 OUTLINE OF THE METHOD

To evaluate the performance of pigment with respect to colour, tinting strength, colour difference (mass tone) and ease of dispersion against approved pigment sample

C-2 APPARATUS

C-2.1 Vibroshaker — 25 degree to 30 degree angular oscillation @ 640 / 660 spm (shakes per minute) simultaneously

C-2.2 Closed SS metal container —125 ml

C-2.3 Glass Beads — diameter 2.5 mm to 3.0 mm

C-2.4 Electronic Balance — accuracy 1 mg

C-2.5 Spatula

C-2.6 Mechanical Stirrer

C-2.7 Black and White Paper — Charts, all of same size and measuring at least 100 x 200 mm, printed and varnished to give adjacent black and white areas readily wetted by, but impervious to solvent or water-thinned paints. The black and white areas shall each be of dimensions not less than 80 x 80 mm. The reflectance of white areas of the cards shall be 80 ± 2 percent when measured over a white tile using a spectrophotometer complying with **C-5.4.2** and that of the black areas shall be not greater than 1 percent, unless otherwise agreed.

To avoid errors due to-variation, from one batch of charts to another, the charts used for a given test should come from the same batch.

C-2.8 Film Applicator — A film applicator giving a uniform film of maximum wet thickness of 80 μm is required. The film laid down shall be at least 70 mm wide, with areas of dimensions not less than 60 x 60 mm and of uniform thickness over both black and white areas of the card. The application of uniform films is facilitated by the use of automatic applicators.

C-3 PROCEDURE

C-3.1 Method of Preparation of the Pigment Concentrate:

Table 2 gives a formulation for 100% completion. For this test, apply factor of 0.4 to all the materials given in Table 2 and weigh the ingredients accordingly. Add 60 g of glass beads in a closed stainless steel (SS) metal container of volume 125 ml. The dispersion time after addition of materials mentioned in Stage 1 is 40 min. After dispersion is complete, add materials mentioned in Stage 2 and run for another 10 min. Collect the pigment concentrate and proceed for the testings.

Table – 2 Guideline Formulation of Preparation of Pigment Concentrate

(Clause 5.2)

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% solids in xylene, Viscosity of solution at 25°C 8.0 – 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)			
€Use glass beads as grinding media 60 g			

C-4 DETERMINATION OF EASE OF DISPERSION:

To assess the ease of dispersion/fineness of grinding by gauge follow the procedure as given in IS 354 (Part 2).

C-5 DETERMINATION OF COLOUR DIFFERENCE (MASS TONE):

C-5.1 Colour Difference (Mass Tone)

C-5.1.1 To check the colour difference of pigment concentrate it needs 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.

C-5.1.2 Catalyst — Medium viscosity polyamide hardener

C-5.1.3 Amine Value — (300 ± 20) mg KOH/g

C-5.2 Calculation of Mixing Ratio:

Epoxy equivalent of Epoxy resin	=	E
Solid content of Epoxy resin	=	X percent
Loading of Epoxy resin in formulation (<i>see</i> Table 2)	=	Y percent
Active content	=	$\frac{Y \times X}{100}$
Amine value of catalyst	=	C
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
$\frac{Y \times X}{100}$ Epoxy cured with		$\frac{56100 \times Y \times X}{100 \times C \times E}$ g Amine

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

Mixing Ratio of Pigment concentrate:

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

C-5.3 Procedure

Mix the pigment concentrate and catalyst as per ratio given in C-5.2 and mix uniformly with a spatula by hand mixing with minimum foam generation. Make drawdowns side by side (approved sample/as agreed between parties vs. sample under evaluation) with the help of film applicator. 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/as agreed between parties in Spectrophotometer (*see* C-5.4).

C-5.4 Degree of Colour Change

C-5.4.1 The colour of the test panel shall be compared against the stipulated shade as given in IS 5 or any agreed standard sample between purchaser and supplier. The colour difference between a pair of painted panels shall be calculated in terms of ΔE (Delta E) by using CMC (2:1) colour difference equation. From spectral measurement, QC program (Color quality control software) calculate CIE (“Commission Internationale de l’Éclairage” or International Commission on Illumination) colour specifications- L* a* b* C* h* based on daylight illumination, as described in C-5.4.2.

C-5.4.2 A reflectance spectrophotometer is used for reflectance measurement. Like Visual Assessment requirement, the sample should be sufficiently large enough to measure using Large Area of View (LAV). Small sized samples measured using Small Area of View (SAV) may not have high repeatability. Sample size should be minimum 2 x 2”. The spectral measurement should be carried out using a reflectance spectrophotometer having following features:

Repeatability – Short-Term : Better than 0.15 ΔE_{CIELAB}

Inter-Instrument Agreement : Better than 0.25 ΔE_{CIELAB}

Wavelength Range : 400-700 nm (360-780 nm preferable)

Wavelength Increment : 10 nm

C-6 DETERMINATION OF TINTING STRENGTH:

C-6.1 To check the tinting strength percent 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.

Table 3
(Clause C-6.1)

Reduced tone and Strength percent [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

C-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 (<i>see</i> Table 3)	=	Y percent
Active content	=	$\frac{Y \times X}{100}$
Amine value of catalyst	=	C
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
$\frac{Y \times X}{100}$ Epoxy cured with		$\frac{56100 \times Y \times X}{100 \times C \times E}$ g Amine
Therefore, 100 g Pigment concentrate cured with		$\frac{561 \times Y \times X}{C \times E}$ g Amine

Mixing Ratio of Pigment concentrate:

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

C-6.3 Procedure

Mix the pigmented base and catalyst as per ratio given in **C-6.2** and mix uniformly with a spatula by hand mixing with minimum foam generation. Make drawdowns side by side (approved sample/as agreed between parties vs. sample under evaluation) with the help of film applicator.

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 tinting strength percent of the specimen against approved sample/as agreed between parties by Spectroscopic method (*see C-6.3.2*).

C-6.3.2 Measure the tinting strength of sample with the spectrophotometer (*see C-5.4.2*). The relative tinting strength can be calculated by taking ratio of the absorbance index of the test sample to that of an agreed reference pigment, expressed as a percentage.

ANNEX D

[Table 1, Item No. (viii)]

TEST FOR FASTNESS TO LIGHT

D-1 GENERAL

D-1.1 Outline of Method — Painted panels made from material and approved sample/as agreed between parties are exposed half covered to carbon arc lamp radiation for specific period and examined for fade in colour.

D-2 APPARATUS

D-2.1 Light Source — artificial light source of carbon arc type.

D-3 PROCEDURE

D-3.1 Prepare a paste of the material as prescribed in IS 33 and apply the paste uniformly and evenly on to a colourless glass plate. Allow the film to dry. Similarly prepare a film of the material using approved sample. Protect half of the portion of the painted glass panels from light by opaque material like aluminium foil. Arrange the glass panels at a distance of 250 mm from the carbon arc lamp. Maintain the temperature of the atmosphere surrounding the specimens throughout the period of exposure between 25 °C to 50 °C. Expose the film to light for 20 hours and then examine for any change in colour. If no appreciable change is noticed, another cycle of 20 hours is given but to the maximum of 3 cycles. At the end of the period, compare the colour of the panels and with other halves also.

D-3.2 The material shall be deemed to have passed the test if fading with the material under test is not more than of the approved sample.

ANNEX E

[Table 1, Item No. (ix)]

TEST FOR SOLUBLE ORGANIC COLOURING MATTER

E-1 GENERAL

E-1.1 Outline of Method — To a boiling solution of the material in ethanol, sodium hydroxide solution and acetic acid are added separately and examined for any development of colour.

E-2 REAGENTS

E-2.1 Ethyl Alcohol — 95 percent (v/v), conforming to IS 323.

E-2.2 Acetic Acid — 10 percent (v/v).

E-2.3 Sodium Hydroxide Solution — approximately 4 N.

E-3 PROCEDURE

E-3.1 Add to ethyl alcohol in a beaker, a small quantity of the material and bring to boiling. Divide the boiling solution into two parts and take in two test tubes. To the test tubes, add a few ml of acetic acid and sodium hydroxide respectively. Observe the colour of the liquid in the test tubes.

E-3.2 The material shall be deemed to have passed this test if the liquids remain colourless.