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BUREAU OF INDIAN STANDARDS

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Draft Indian Standard

CHLORINATED RUBBER FOR PAINTS — SPECIFICATION

(First Revision of IS 13467)

(ICS 87.060.10)

Raw materials for Paints, Varnishes and Related Product Sectional Committee, CHD 21	**Last Date for Comments: 6th March 2024**

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

FOREWORD

(Formal Clause shall be added later)

Chlorinated rubber is used principally in protective paints and coating where resistance to chemicals, corrosive atmosphere, presence of a large amount of moisture, daily decontamination of surface by washing, and even continuous immersion is required.

This standard was originally published in 1992. This revision has been taken to bring out the standard in the latest style and format of the Indian Standard. In addition, the following changes have been made:

a) The U-tube viscometer test method has been replaced with the Brookfield viscometer due to its inefficiency and time-consuming. Similarly, the bomb calorimetry method has been replaced by the combustion method, due to outdated methodology of bomb calorimetry.

b) To address health and safety concerns, restrictions for lead has been tightened and restriction for toxic heavy metals has been introduced;

c) A suitable precautionary note has been added in the marking clause in order to prevent unforeseen events, and;

d) 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

CHLORINATED RUBBER FOR PAINTS –SPECIFICATION

(*First Revision*)

1 SCOPE

This standard prescribes the requirements and the methods of test for the material commercially known as chlorinated rubber for paints.

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 GRADES

The material shall be of the following three grades depending upon its viscosity:

a) Grade 10,

b) Grade 20, and

c) Grade 40.

The grade number is the nominal viscosity of 20 percent solution in centipoise of the grade.

4 REQUIREMENTS

4.1 Description

The material shall be white to off white colour powder and free from foreign matter.

4.2 Lead Restriction

The material shall be tested for restriction from lead in accordance with 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 (*see* Note).

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

4.3 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.

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

Table 1 Requirements for Chlorinated Rubber for Paints

(*Clause* 4.4)

SL NO.	CHARACTERISTIC	REQUIREMENT FOR CHLORINATED RUBBER			METHOD OF TEST, REF TO ANNEX B
		Gr l0	**Gr 20**	**Gr 40**	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Volatile matter, percent by mass, 60 °C, 1 h under vacuum, *Max*	2.0	2.0	2.0	**B-2**
ii)	Viscosity* a) 20 percent solution (m/m), centipoise b) 30 percent solution (m/m), sec	 (10 ± 2) (45 ± 5)	 (20 ± 2) (70 ± 5)	 (40 ± 4) (140 ± 10)	**B-4** **B-4.1** **B-4.2**
iii)	Clarity of the film	Shall be clear and free from bits			**B-5**
iv)	Colour, *Max*	12	12	12	**B-6**
v)	Chlorine, percent by mass, *Min*	60.0	60.0	60.0	**B-7**
vi)	Free chlorine	— To pass the test —			**B-8**
vii)	Ash, percent by mass, *Max*	0.50	0.50	0.50	**B-9**
viii)	Acidity (as HCI), percent by mass, *Max*	0.05	0.05	0.05	**B-10**
ix)	Iron content, ppm, *Max*	10	10	10	**B-11**
* Either (a) or (b) method can be used. However, in case of dispute, method (a) would be the referee method.					

5 KEEPING PROPERTY

The material, when stored in normal conditions for 12 months, shall retain its original properties and conform to requirements given in **4.**

6 PACKING AND MARKING

6.1 Packing

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

6.2 Marking

6.2.1 The containers shall be marked with the following information:

a) Name of the material;

b) Net mass of the material;

c) Batch No. or Lot No. in code or otherwise; and

d) Month and year of manufacture

e) Expiry date/shelf life/best before

f) Lead content (Maximum);

g) Toxic heavy metals content

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

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

7 SAMPLING

Representative samples shall be drawn as prescribed in Annex C.

ANNEX A

(*Clause* 2)

LIST OF REFERRED INDIAN STANDARDS

IS No	*Title*
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 (*fifth revision*)
IS 264 : 2005	Nitric acid — Specification *(third revision)*
IS 354 (Part 1) : 1987	Method of sampling and test for resins for paints : Part 1 General test methods *(second revision)*
IS 1070 : 2023	Reagent grade water — Specification (*fourth revision*)
IS 2263 : 1979	Methods of preparation of indicator solutions *(first Revision)*
IS 2316 : 1990	Methods of preparation of standard solutions for volumetric analysis (*first revision)*
IS 2630 : 2021	Nitrobenzene — Specification (*third revision*)
IS 4905 : 2015 ISO 24153: 2009	Random sampling and randomization procedures (*first revision*)
IS 15556 : 2005	Volumetric and spectrophotometric estimation of iron

ANNEX B

(*Table* 1)

REQUIREMENTS FOR CHLORINATED RUBBER FOR PAINTS

B-l 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 the analysis.

B-2 VOLATILES, PERCENT BY MASS

B-2.1 Apparatus

B-2.1.1 *Petri-Dish* — Approximately 7 cm to 8 cm diameter and 2 cm deep.

B-2.1.2 *Vacuum Oven* — Provided with temperature indicator and control, vacuum regulating device, and capable of maintaining recommended temperature and vacuum.

B-2.1.3 *Vacuum Pump*

B-2.2 Procedure

Weigh accurately about 1 g to 1.5 g of the material and spread uniformly in a petri-dish. Put the petri-dish in vacuum oven maintained at 60 °C and a pressure of about 133.322 Pa. After one hour remove the petri-dish and keep in the desiccator. Allow it to cool to room temperature and reweigh. Calculate loss of mass.

NOTE — 1 torr = $\frac{1}{760}$ atm =13 332.2 Pa

B-2.3 Calculation

Volatile matter,

$$\text{percent by mass} = \frac{M_1 - M_2}{M_1} \times 100$$

where,

M_1 = initial mass of the sample, and

M_2 = final mass of the sample.

B-3 PREPARATION OF SOLUTION OF CHLORINATED RUBBER IN O-XYLENE

B-3.1 Reagent- O-xylene

B-3.2 Procedure

Prepare 30 percent *m/m* solution of chlorinated rubber in o-xylene by weighing accurately 60.0 g of the sample in 500 ml beaker and adding 140 g of o-xylene in it. Stir with glass rod. Note the total mass of the beaker and warm on water bath or hot plate. Stir well till whole mass dissolves in the xylene. Solution should be clear. Reweigh the beaker and compensate for loss of solvent. Store the solution in stoppered bottle and use it for tests prescribed in **B-4**, **B-5**, **B-6** and **B-8**.

B-4 DETERMINATION OF VISCOSITY

B-4.1 Brookfield Viscometer Method

Determine the viscosity of 20 percent (*m/m*) solution in toluene as per the method mentioned in IS 354 (Part 1) using Brookfield viscometer and report the result.

B-4.2 Flow Cup (Ford Cup Viscometer No. 4)

Determine the viscosity of 30 percent (*m/m*) solution, as prepared in **B-3**, as per the method mentioned in IS 354 (Part 1) using flow cup (ford cup viscometer No. 4) and report the result.

B-5 CLARITY OF THE FILM

Prepare the solution as in **B-3**. Take a drawdown of the 30 percent solution on a clean glass plate with 6 millibar applicator (150 microns). Allow to dry the film in completely dust free room for 30 minutes and observe the clarity of film.

B-6 COLOUR OF 30 PERCENT (W/W) SOLUTION

B-6.1 Prepare solution as in **B-3**. Measure the colour of the solution as per standard test method of colour of transparent liquids using Gardner colour standards {*see* IS 354 (Part 1)}.

B-6.2 Report

Report the colour as the number of the standard, most closely matching the specimen. For most precise measurement report as either matching in standard or lighter, or darker. Thus, for example, colour between 11 and 12, the increasingly darker colour description would be 11, 11+, 12- and 12.

B-7 DETERMINATION OF CHLORINE CONTENT BY COMBUSTION METHOD

B-7.1 Outline of the Method

This method describes the determination of the amount of chlorine content by the combustion or pyrolysis method of chlorinated rubber or compound specimen. The specimen is heated up to maximum of 800 °C for complete decomposition. Halogen acid gas evolved during the pyrolysis of specimen and suitably passed into known strength of sodium hydroxide solution and calculated. This test method is not recommended for use where the amount of halogen acid evolved is less than 0.5 percent.

B-7.2 Apparatus

B-7.2.1 *Tube Furnace* — with thermostatic temperature control up to 1 000°C. The length of the tube furnace shall be at least 100 mm.

B-7.2.2 *Quartz (or Other Suitable) Combustion Tube* — Approx. 20 mm inside dia, 25 mm outside dia, and 700 mm long.

B-7.2.3 *Quartz / Porcelain (or Other Suitable) Combustion Boat* — Approx.76 mm x 10 mm x 9 mm.

B-7.2.4 *Four Wash Bottles* — (55 ± 5) mm. dia

B-7.2.5 *Glass Tubing and Silicone Rubber Stoppers* — Used to connect the wash bottles to the combustion tube. Connection between the wash bottles shall be made using silicone rubber with the glass tubing together in the connection or by using ground glass joints.

B-7.2.6 *Physical Balance* — Accuracy 0.1 mg.

B-7.2.7 *Air Flowmeter* — 0 ml/minute to 200 ml/minute. For set up of apparatus (*see* Fig. 1 or 2).

B-7.3 Reagents

B-7.3.1 *Sodium Hydroxide Solution* — N/10 (0.1 normal).

B-7.3.2 *Nitric Acid Solution* — 6 N.

B-7.3.3 *Silver Nitrate Solution* — N/10 (0.1 normal).

B-7.3.3.1 *Preparation*

Dry finely powdered Analytic Grade (AR) $AgNO_3$ at 150 °C for 2 hours and allow it to cool in a desiccator. Weigh out accurately 8.496 g. Dissolve it in water and make up the volume to 500 ml in a volumetric flask. This gives 0.1 N $AgNO_3$ solutions. The weight of $AgNO_3$ will vary depending upon the purity of $AgNO_3$ used, for example, if this is 99.9 percent pure, then the weight should be multiplied by the purity factor of AR $AgNO_3$ in this concentration:

$$8.496 \times \frac{1}{0.999} = 8.5045\ g$$

B-7.3.4 *Ammonium Thiocyanate Solution* — N/10 (0.1 normal).

B-7.3.4 .1 *Preparation*

Weigh about 9 gm of Analytic Grade (AR) ammonium thiocyanate and dissolve it in 1 liter of distilled water in volumetric flask, Shake well. Standardization of this solution is carried out with 0.1 N $AgNO_3$.

B-7.3.4.2 *Procedure*

Take the above solution of NH_4CNS in a burette (50 ml). Pipette out 25 ml of 0.1 N $AgNO_3$ in a conical flask. Add 5 ml of 6 N HNO_3 and 2 ml of the ferric ammonium sulphate. Ferric alum indicator is prepared by making 40 percent aqueous solution containing few drops of 6 N nitric acid. Run in NH_4NS solution from the burette. At first a white precipitate is produced rendering the liquid milky appearance, as each drop of thiocyanate falls in it, it produces a reddish-brown cloud which quickly disappears on shaking. As the end point approaches, the precipitate becomes flocculent and settles easily. Finally, one drop of the thiocyanate solution produces a faint brown colour, which no longer disappears upon shaking. This is the end point. Note it as X ml.

Now, calculate the normality of ammonium thiocyanate as follows:

$$NH_4NS: AgNO_3$$

$$N_1V_1 = N_2V_2$$

$$N_I \text{ x } (X) = 0.1 \text{ x } 25$$

$$X = \frac{0.1 \times 25}{N_1}$$

Where,

N_1 = normality of NH_4NS

V_1 = volume of NH_4NS used for this titration = X

N_2 = normality of $AgNO_3$

V_2 = volume of $AgNO_3$ (25 ml) taken for standardization.

This (N_1) is to be treated as factor for NH_4NS

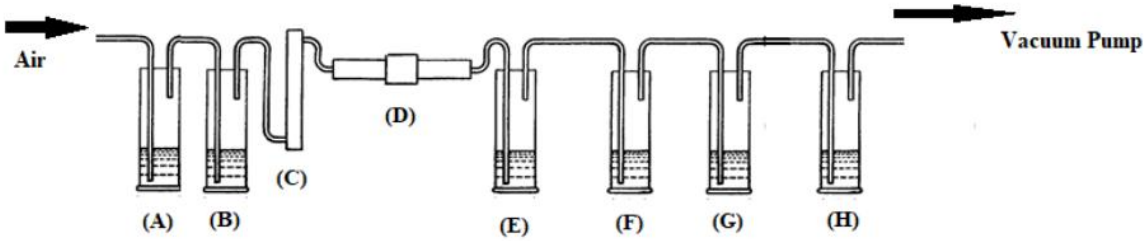
N_1 = Factor = F

B-7.3.5 *Ferric Ammonium Sulphate Solution* — 40 percent**.**

B-7.3.6 *Dry Air*

B-7.3.7 *Potassium Hydroxide* **—** N/10 (0.1 normal).

B-7.3.8 *Sulphuric Acid* **—** N/10 (0.1 normal).



(A) = Wash Bottle KOH

(B) = Wash Bottle H_2SO_4

(C) = Air Flow Meter

(D) = Combustion Furnace

(E) = Wash Bottle with sintered glass filter NaOH

(F) = Wash Bottle with sintered glass filter NaOH

(G) = Wash Bottle with sintered glass filter NaOH

(H) = Wash Bottle with sintered glass filter NaOH

FIG. 1 TEST SET UP

B-7.4 Procedure

B-7.4.1 Weighed 0.1 g to 0.2 g of the specimen in the combustion boat, which shall then be inserted into the combustion tube placed in the tube furnace. In order to reduce condensation in the tube, the exit end of the combustion tube shall not project more than 60 mm from the end of the furnace.

B-7.4.2 The combustion tube shall be connected to the four wash bottles each containing 150 ml of N/10 (0.1 N) sodium hydroxide solution. The dry air shall be passed through the apparatus at a rate of (110 ± 5) ml/minute.

B-7.4.3 The temperature of the tube furnace shall then be raised to 800 °C at a rate of approx. 20°C/minute and maintained at 800 °C for 15 minutes.

B-7.4.4 The three wash bottles shall then be disconnected and when cool, the combustion tube and connecting tubes shall be washed with distilled water and the total volume shall be made up to 500 ml with distilled water. Take 100 ml of the aliquot into Erlenmeyer flask and 2 ml of concentrated nitric acid, 25 ml of 0.1 N Silver Nitrate ($AgNO_3$) and 5 ml of 50 percent solution of ferric ammonium sulphate containing a few drops of 6 N nitric acid added and mixed together.

B-7.4.5 After filtration through on sintered glass crucible to remove precipitated silver chloride the solution shall then be titrated with 0.1 N (N/10) ammonium thiocyanate solution.

B-7.5 Calculation

The amount of chlorine is calculated by using the following formula*:*

$$\frac{3.65 \times (B - A) \times F \times 500}{m \times 100}$$

Where,

B = volume of 0.1 N ammonium thiocyanate solution used in the blank test,

A = volume of 0.1 N ammonium thiocyanate solution used in the determination of sample,

F = factor for 0.1 N ammonium thiocyanate solution, and

m = mass of the sample taken in grams.

B-8 FREE CHLORINE CONTENT

A-8.1 Reagent

B-8.1.1 *Starch-Iodide Solution* **—** Prepare starch iodide solution by dissolving 5.0 g potassium iodide in 100 ml of water containing 10 ml of 0.2 percent starch solution.

B-8.2 Procedure

Transfer 3 to 5 ml of 30 percent *m/m* solution in o-xylene to a test tube (*see* **B-3**), add 2 ml to 3 ml of starch iodide solution and shake vigorously for 1 minute.

B-8.3 Observation

No blue colouration shall be observed in the aqueous layer.

B-9 ASH CONTENT

B-9.1 Apparatus

B-9.1.1 *Silica Crucible* **—** 50 ml capacity.

B-9.1.2 *Muffle Furnace* — Capable of maintaining temperature up to (750 ± 20) °C with temperature indicator and control.

B-9.2 Procedure

Ignite a silica crucible, cool in desiccator and weigh to the nearest 0.1 mg. Weigh accurately about 15 g to 20 g of the sample in the silica crucible. Place it on a tripod stand and heat gently with a Bunsen burner until the sample catches flame. Remove the burner and allow the sample to burn completely. Reheat from time to time to bring about complete combustion. Carry out this heating in hood having good exhaust system. Remove the crucible and place it in a furnace adjusted at a temperature (750 ± 20) °C until free from carbon. When ashing is complete, cool the crucible in a desiccator and then weigh it to the nearest 0.l mg. Continue with the ignition until constant mass is obtained.

B-9.3 Calculation

Calculate the ash content as follows:

$$\text{Ash content, percent by mass} = \frac{M_1 - M}{M_2} \times 100$$

where

M_1 = mass of crucible and ash,
M = mass of the empty crucible, and
M_2 = mass of the sample.

B-10 ACIDITY

B-10.1 Outline of the Method

Inorganic acids are allowed to dissolve in water and estimated by titration against standard alkali.

B-10.2 Reagents

B-10.2.1 *Standard Sodium Hydroxide Solution,* 0.1 N (*see* IS 2316)

B-10.2.2 *Phenolphthalein Indicator*

Three percent solution in 50 percent aqueous ethanol. (*see* IS 2263)

B-10.3 Procedure

Place 10 g to 15 g of the sample weighed to the nearest 0.05 g in a 250 ml flask with 100 ml of boiled and cooled distilled water. Allow it to soak for 1 hour at room temperature swirling at 10 minutes intervals. Filter the contents of the flask in 250 ml standard volumetric flask through filter paper No. 41. Give washings and dilute up to the mark. Pipette out 50 ml of diluted solution in Erlenmeyer flask. Add 2 drops of phenolphthalein indicator and titrate to a faint pink end point with 0.1 N standard sodium hydroxide solution.

B-10.4 Calculation

Acidity (as HCI),

$$\text{percent by mass} = \frac{V \times N \times 18.25}{M}$$

where,

V = Volume of 0.1 N NaOH required for titration, in ml;

N = normality of NaOH solution; and

M = weight of the sample taken.

B-11 IRON CONTENT (COLORIMETRIC METHOD)

Convert 15 g to 20 g of sample into ash by ash content method (*see* **B-9**). Dissolve this ash in concentrated hydrochloric acid and dilute the solution to appropriate volume and determine the iron content by calorimetric method as prescribed in IS 15556.

ANNEX C

(*Clause* 7)

SAMPLING PLAN OF CHLORINATED RUBBER FOR PAINTS

C-1 LOT

The bags of chlorinate rubber of the same grade belonging to the same batch of production shall constitute a lot. Each lot shall be tested separately.

C-2 SCALE OF SAMPLING

The number of bags to be selected for sampling shall be as given in Table 2.

These bags shall be selected at random from the lot, as prescribed in IS 4905.

C-3 PREPARATION OF TEST SAMPLES

From each selected bag, a representative portion of material shall be taken, which shall be sufficient for carrying out tests for all characteristics given in the specification.

C-3.1 Composite Sample

Out of these portions, a small but equal quantity of material shall be taken and mixed thoroughly to make a composite sample.

C-3.2 Individual Sample

The remaining portion of material from each selected bag shall constitute an individual test sample.

C-4 Number of Tests

C-4.1 Test for **4.1** (Description) and Sl No. (v) of Table 1 (Chlorine content) shall be performed on the individual sample (**C-3.2**). The tests for all other characteristics of **4.2** shall be conducted on the composite sample (**C-3.1**).

C-5 Criteria for Conformity

The lot shall be deemed to conform to the specification if all the test results according to **C-4.1** satisfy the corresponding requirements.

Table 2 Scale of Sampling

(*Clause* C-2)

Number of Bags in the Lot	Number of Bags to be Selected

N	*n*
(1)	(2)
up to 50	3
51 to 100	4
101 to 200	5
201 to 300	6
301 and above	7