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Indian Standard
SPECIFICATION FOR
TITANIUM DIOXIDE, RUTILE FOR PAINTS

( First Reprint OCTOBER 1997 )

UDC 667.632.118.22

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

Gr 7 September 1981
Indian Standard

SPECIFICATION FOR
TITANIUM DIOXIDE, RUTILE FOR PAINTS

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(Continued on page 2)
AMENDMENT NO. 1 FEBRUARY 1993
TO
IS 9788 : 1981 SPECIFICATION FOR TITANIUM DIOXIDE, RUTILE FOR PAINTS

( Page 5, clause 4.2 ) — Insert the following new clause after 4.2:

'4.2.1 The rule content shall be 99 percent, Min, of the coal titanium dioxide content.'

( Page 15, clause C-1.1, last line ) — Substitute '0.318' for '31.8'.

( CHD 021 )
IS : 9788 - 1981

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(Continued on page 26)
Indian Standard
SPECIFICATION FOR
TITANIUM DIOXIDE, RUTILE FOR PAINTS

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 27 February 1981, after the draft finalized by the Raw Materials for Paints Industry Sectional Committee had been approved by the Chemical Division Council.

0.2 Titanium dioxide is one of the most important pigments used in paint industry. The material is mainly of two types, namely, anatase and rutile and both are used substantially in paint industry. Though both types of pigment are chemically similar, they differ in crystalline structure and hence in physical properties. The type is identified more accurately by X-ray defraction than the relative density. IS - 411-1968* covered both anatase and rutile varieties. In view of distinct usages of the two types of this important pigment, the concerned technical committee decided to publish standards pertaining to them separately. The second revision of IS : 411 would now cover only anatase grade and rutile grade is covered in this standard.

0.3 In this standard, requirements for dispersibility on high speed disperser; durability; special properties, such as, opacity, gloss and gloss retention, ease of dispersion, resistivity of aqueous extract, settling property; and water demand of the material which were not covered earlier have been prescribed. Method for determination of mass colour of the material based on formulation for air drying alkyd gloss paint, exterior emulsion paint and stoving paint has been included. For determination of reducing power the method based on the use of phthalocyanine green pigment instead of conventional use of ultramarine blue pigment has been prescribed and the tinting strength obtained by this method has been designated as 'ISI Tinting Strength.'

0.3.1 Besides, the titanium dioxide, rutile, manufactured by chloride or sulphate process, has been classified in three grades so as to suit the different end uses of the pigment in paint industry. The modern rutile pigment is characterized by the type, amount and manner of deposition of surface treatment compounds. The manufacturers may indicate the

*Specification for titanium dioxide for paints (first revision)
type of morgana and organic treatment given. To allow for this, and for convenience, the grades are classified according to titanium dioxide content.

0.4 In the preparation of this standard, substantial assistance has been derived from data supplied by M/s Asian Paints (I) Ltd, Bombay, Goodlass Nerolac Paints Ltd, Bombay and the Indian Paint Association, Calcutta which is thankfully acknowledged. This standard also achieves substantial alignment with rutile grades specified in ISO 591-1977 'Titanium dioxide for paints' issued by the International Organization for Standardization (ISO).

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, 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.

1. SCOPE

1.1 This standard prescribes requirements and methods of sampling and test for rutile titanium dioxide used as a pigment in paint industry.

2. TERMINOLOGY

2.1 For the purpose of this standard, definitions of terms (including approved sample), given in IS 1903-1963† and under 2 of IS : 33-1976‡ shall apply.

3. GRADES

3.1 The material shall be of the following three grades depending on surface treatment and titanium dioxide content:
   a) Grade R₁,
   b) Grade R₂, and
   c) Grade R₃

3.1.1 The manufacturers shall declare the modifiers used for surface treatment of rutile titanium dioxide.
4. REQUIREMENTS

4.1 Description — The material shall be a soft, dry powder and shall consist essentially of titanium dioxide of rutile crystalline structure as determined by X-ray examination.

4.2 Composition — The material after drying to a constant mass at 100 ± 2°C, when determined as prescribed in Appendix A, shall contain titanium dioxide as specified in Table 1.

4.3 Resistance to Chalking — When tested as prescribed in Appendix B, the time taken for the commencement of chalking in a paint film incorporating the material shall be not less than that of a paint film incorporating the approved sample prepared and tested in the same manner, at the same time and by the same person.

4.4 Dispersibility — The material when tested as prescribed in Appendix C shall pass the test and shall be not inferior (in reverting of finish or reagglomeration) to that of the approved sample.

4.5 Durability — The material, when tested as prescribed in Appendix D, shall pass the test.

4.6 Special Properties — The material shall have ease of dispersion, gloss and gloss retention and opacity similar to those of the approved sample of paint prepared as given in E-1.2 and when tested as prescribed in 7.6, 7.7 and 10 of IS: 101-1964*, respectively.

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

5. PACKING AND MARKING

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

5.2 Marking — The packing shall be securely closed and marked with the following particulars:

a) Name, grade and the modifier(s) used in surface treatment;
b) Name of the manufacturer or his recognized trade-mark, if any;
c) Mass of the material;
d) Batch No. or lot No. in code or otherwise; and
e) Month and year of manufacture.

*Methods of test for ready mixed paints and enamels (second revision)
### TABLE 1 REQUIREMENTS FOR TITANIUM DIOXIDE, RUTTLE, FOR PAINTS

*(Clauses 4.2 and 4.7)*

<table>
<thead>
<tr>
<th>SL No</th>
<th>CHARACTERISTIC</th>
<th>REQUIREMENT FOR GRADE</th>
<th>METHOD OF TEST, REF- TO</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>R₁</td>
<td>R₂</td>
</tr>
<tr>
<td>(1)</td>
<td>(2)</td>
<td>(3)</td>
<td>(4)</td>
</tr>
<tr>
<td>i)</td>
<td>Titanium dioxide (TiO₂) content, percent by mass, Min</td>
<td>97</td>
<td>90</td>
</tr>
<tr>
<td>ii)</td>
<td>Mass colour</td>
<td>Close match to the approved sample</td>
<td>E</td>
</tr>
<tr>
<td>iii)</td>
<td>Reducing power</td>
<td>Not inferior to the approved sample</td>
<td>F</td>
</tr>
<tr>
<td>iv)</td>
<td>Volatile matter, percent by mass, Max</td>
<td>0.5</td>
<td>0.5</td>
</tr>
<tr>
<td>v)</td>
<td>Matter soluble in water, percent by mass, Max</td>
<td>0.5</td>
<td>0.5</td>
</tr>
<tr>
<td>vi)</td>
<td>Oil absorption†</td>
<td>18 ± 3</td>
<td>20 ± 4</td>
</tr>
<tr>
<td>vii)</td>
<td>pH of aqueous suspension at 27°C</td>
<td>← 6 to 8 →</td>
<td>—</td>
</tr>
<tr>
<td>viii)</td>
<td>Residue on 45 microns sieve, percent by mass, Max</td>
<td>0.1</td>
<td>0.1</td>
</tr>
<tr>
<td>ix)</td>
<td>Resistivity of aqueous extract</td>
<td>— ±5 percent of the approved sample</td>
<td>G</td>
</tr>
<tr>
<td>x)</td>
<td>Water demand</td>
<td>— ±5 percent of the approved sample</td>
<td>H</td>
</tr>
<tr>
<td>xi)</td>
<td>Settling property</td>
<td>← To pass the test →</td>
<td>J</td>
</tr>
</tbody>
</table>

*Methods of sampling and test for inorganic pigments and extenders for paints (second revision).
†However, it may be within ± 10 percent of the approved sample, if any
5.2.1 The packages may also be marked with the Standard Mark.

5.7.2 The use of the Standard Mark is governed by the provisions of the 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.

6. SAMPLING

6.1 Representative samples of the material shall be drawn as prescribed under 4 of IS : 33-1976*.

6.2 Criteria for Conformity — A lot shall be declared as conforming to the requirements of this standard, if test results of the composite sample — satisfy the requirements prescribed in 4.

7. TEST METHODS

7.1 The tests shall be conducted as prescribed in IS : 33-1976*, and Appendices A to J. Reference to IS : 33-1976* is given in col 7 of Table 1 and to appendices in 4.2 to 4.5 and col 6 of Table 1

7.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water ( see IS . 1070-1977† ) shall be employed in tests.

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

APPENDIX A

[ Clause 4.2, and Table 1, Item (i) ]

DETERMINATION OF TITANIUM DIOXIDE CONTENT

A-0. GENERAL

A-0.1 Two methods for determination of titanium dioxide have been prescribed. For routine testing, either Method 1 or Method 2 may be followed, but in case of dispute, Method 1 shall be followed.

*Methods of sampling and test for inorganic pigments and extenders for paint ( second revision ).

†Specification for water for general laboratory use ( second revision )
A-1. METHOD 1: ZINC REDUCTION METHOD

A-1.0 Outline of Method — A solution of the dry pigment is made with concentrated sulphuric acid and ammonium sulphate. Titanium is reduced with zinc amalgam in Nakazono reductor and the resulting material is titrated with ferric alum.

A-1.1 Reagents

A-1.1.1 Concentrated Sulphuric Acid — Relative density 1.84 (see IS 266-1977*).

A-1.1.2 Ammonium Sulphate

A-1.1.3 Zinc Amalgam — 3 percent prepared as given in A-1.1.3.1.

A-1.1.3.1 Place 50 ml of mercury in a small porcelain dish on a hot water bath covering the surface of the mercury with 2 percent (m/v) sulphuric acid. Add 25 g of zinc in small granules. Stir from time to time and replenish the dilute acid with water as required. When all zinc has disappeared, allow the amalgam to cool and stand for several hours. Filter through a Gooch crucible with no asbestos pad. Store the amalgam in a glass bottle under 2 percent (m/v) sulphuric acid.

A-1.1.4 Standard Ferric Ammonium Sulphate Solution — 0.0625 N standardized as given in A-1.1.4.1.

A-1.1.4.1 Transfer by pipette 50 ml of the ferric ammonium sulphate solution to be standardized to the Nakazono reductor prepared ready for use as described in A-1.3.2. Displace the air in the reductor by passing carbon dioxide for 3 minutes. Close the taps B and C. Bring the amalgam into intimate contact with ferric ammonium sulphate solution by shaking the apparatus vigorously for 5 minutes. Place the apparatus on a mg stand. Dram the amalgam and pass inert gas as prescribed under A-1.3.4. Titrate this against standard potassium permanganate solution. The end point is the appearance of light pink colour persisting for at least 30 seconds. Calculate the strength of ferric ammonium sulphate solution as follows:

\[
\text{Strength of ferric ammonium sulphate solution} = \frac{V \times 0.1}{50}
\]

where

\[V = \text{volume in ml of 0.1 N potassium permanganate solution required for titration}\]

*Specification for sulphuric acid (second revision).
A-1.5 Potassium Thiocyanate Solution — 10 percent (m/v).

A-1.2 Nakazono Reductor — The apparatus as shown in Fig. 1, consists of a bulb A with a capacity of 500 ml with three stopcocks B, C and D attached, to the stopcock D may be attached by means of thick rubber tubing, E, a small cylindrical flask F, of about 50 ml capacity. The stopcock C which is smaller in bore than either of the other two, serves to admit carbon dioxide or nitrogen.

A-1.3 Procedure

A-1.3.1 Weigh approximately 0.5 g of the dried material to the nearest 0.1 mg, into a 250-ml beaker. Add 20 ml of concentrated sulphuric acid
and 10 g of ammonium sulphate. Mix carefully and heat on a hot plate to fuming and over strong flame until dissolution is complete, observing caution while examining the solution. Cool the solution, dilute with 100 ml of water, stir and filter, if necessary.

**A-1.3.2** Open stopcock $C$, transfer sufficient quantity of 4 percent sulphuric acid to fill the bottom flask $F$ and leave no air space below the stopcock $D$. Close stopcock $D$ and add 20 ml of zinc amalgam to $A$. The reductor is now ready for use.

**A-1.3.3** To the amalgam contained in $A$ transfer the solution, warmed to 50°C, which shall not exceed 300 ml in volume. Then pass carbon dioxide or nitrogen for about 3 minutes. Close taps $B$ and $C$ and simultaneously disconnect the carbon dioxide supply. Bring the amalgam into intimate contact with the solution by shaking the apparatus vigorously for 5 minutes, holding in such a way that the stopcocks are kept shut and in position. The rate of reaction is apparent from the development of the characteristic violet colour of the trivalent titanium.

**A-1.3.4** Place the apparatus on a ring stand. Open stopcock $D$ slightly and allow the amalgam to flow slowly into $E$. Close $D$ immediately as soon as the amalgam has been completely transferred. With taps $B$ and $D$ open, pass inert gas through $D$ as before, add 10 ml of potassium thiocyanate solution and titrate with standard ferric ammonium sulphate solution, until a faint pink colouration persists for one minute.

**A-1.3.5** Facilitate the titration by attaching a piece of very narrow glass tubing to the tip of the burette with a piece of rubber tubing. The extension passes through stopcock $B$ into the bulb $A$.

**A-1.4 Calculation**

**A-1.4.1** Calculate the titanium dioxide content of the material as follows:

$$\text{Titanium dioxide content, percent by mass} = \frac{V \times 0.005 \times 100}{M}$$

where

$V =$ volume in ml of standard ferric ammonium sulphate solution, and

$M =$ mass in g of the pigment taken.

**A-2. Method 2: Aluminium Reduction Method**

**A-2.0 Outline of the Method** — This method is similar to zinc reduction method, but easier and quicker to operate. Titanium is reduced to trivalent state by aluminium and titrated against ferric ammonium sulphate using ammonium thiocyanate as indicator.
A-2.1 Apparatus

A-2.1.1 Delivery Tube — made of 4 mm internal diameter glass tube bent in such a way as to have a horizontal run of about 150 mm and vertical drop of about 75 mm at one end, and a vertical drop of about 150 mm at the other end.

A-2.1.2 Long Necked Flask — 500 ml capacity.

A-2.2 Reagents

A-2.2.1 Ammonium Sulphate — ( see IS : 826-1967* ).

A-2.2.2 Sulphuric Acid — relative density 1.84 ( see IS : 266-1977† ).

A-2.2.3 Hydrochloric Acid — relative density 1.19 ( see IS : 265-1976‡ ).

A-2.2.4 Aluminium Foil — electrolytic grade.

A-2.2.5 Sodium Bicarbonate Solution — saturated.

A-2.2.6 Ammonium Thiocyanate Indicator Solution — Prepare by dissolving 24.5 g of ammonium thiocyanate in 80 ml of hot water, filter and cool to room temperature, dilute to 100 ml and preserve in a well stoppered amber coloured bottle.

A-2.2.7 Standard Ferric Ammonium Sulphate Solution — Prepare by dissolving 30.16 g of fresh ferric ammonium sulphate in 800 ml of water containing 15 ml of concentrated sulphric acid ( see IS : 266-1977† ). Add 0.1 N potassium permanganate solution until a very slight pink colour is obtained. Dilute to 1 000 ml and mix thoroughly. Filter if necessary. Standardize with 0.190 to 0.210 g of standard titanium dioxide as prescribed in A-2.3 and calculate titanium dioxide equivalent of solution in g of titanium dioxide per ml of solution as given below:

Titanium dioxide equivalent = \( \frac{M \times P}{V \times 100} \)

where

- \( M \) = mass in g of standard titanium dioxide ( TiO\(_2\) ) taken for test,
- \( P \) = percent of TiO\(_2\) in standard titanium dioxide used, and
- \( V \) = volume in ml of ferric ammonium sulphate solution required for titration.

*Specification for ammonium sulphate, fertilizer grade ( first revision )
†Specification for sulphuric acid ( second revision )
‡Specification for hydrochloric acid ( second revision )
A-2.3 Procedure

A-2.3.1 Weigh accurately 0.190 to 0.210 g of dry material and transfer the same to a 500-ml long necked flask. Add 7 to 9 g of ammonium sulphate and 20 ml of sulphuric acid. Mix carefully. Heat on a hot plate to fuming and over a strong flame until dissolution is complete. Cool and add 120 ml of water and 20 ml of hydrochloric acid. Boil the contents and remove from heat.

A-2.3.2 Insert the short end of the delivery tube into one hole of a two-hole rubber stopper suitable for the long necked flask. Insert a glass rod with a hook at the bottom end in the other hole in such a way that the bottom end shall be nearer to the bottom of the flask when the stopper is fitted into the flask. Attach 1 g of aluminium foil to the bottom end of the rod by coiling it around the rod. Insert the stopper carrying the rod with foil and delivery tube into the flask in such a way that the foil is near the bottom of the flask and at the same time the long end of the delivery tube is near the bottom of a 250-ml beaker containing 150 ml of sodium bicarbonate solution.

A-2.3.3 Heat the flask as soon as the dissolution of aluminium is completed to gentle boiling for 3 to 5 minutes without disturbing the assembly. Cool to 60°C by partial immersion in water and siphon the sodium bicarbonate solution into the flask giving an atmosphere of carbon dioxide over the reduced titanium solution. Withdraw the stopper, rinse the glass water, catching the rinsings in the flask. Add 2 ml of ammonium thiocyanate indicator solution and titrate immediately against standard ferric alum solution to a straw coloured end point.

A-2.4 Calculation

A-2.4.1 Calculate the percentage of titanium dioxide ($\text{T}_1\text{O}_2$) as follows

$$\text{T}_1\text{O}_2\text{ content, percent by mass} = \frac{V \times E \times 100}{M}$$

where

- $V =$ volume in ml of ferric ammonium sulphate solution used in the test,
- $E =$ titanium dioxide equivalent (see A-2.2.7) of ferric ammonium sulphate solution in g/ml, and
- $M =$ mass in g of material taken for the test.
APPENDIX B

( Clause 4.3 )

DETERMINATION OF RESISTANCE TO CHALKING

B-0. GENERAL

B-0.1 Outline of the Method — The pigment under test and the approved sample are converted into paints, applied to separate test panels and subjected to outdoor exposure. A piece of suitable cloth is drawn across the surface of the film under specific conditions and the surface examined for any chalking.

B-1. PREPARATION OF THE PAINT

B-1.1 Paints of the sample under test and the approved sample shall be prepared as given in E-1.2, E-1.3 and E-1.4.

B-2. PREPARATION OF THE TEST PANEL

B-2.1 Apply the two paints vertically side by side on the same panel which shall be of wood or of any other material, agreed to between the purchaser and the supplier and give identical outside exposure at an angle of 45° facing South. The area of each paint exposed shall be not less than 120 cm². Determine the time taken for chalking to commence by fortnightly examination of the panel by the procedure prescribed under B-3.

B-3. PROCEDURE

B-3.1 Draw a piece of black cloth, such as velvet, silesian, italian or some other suitable cloth of approximately 25 × 100 mm size, under a load of 100 g applied through a disc of soft rubber of 25 mm diameter and about 3.2 mm thick, over the surface of the paint for a distance of 50 mm in approximately one second while the panel rests in a horizontal position.

B-4. OBSERVATION

B-4.1 Chalking shall be considered to have commenced when the surface of the black cloth is just perceptibly marked white.
APPENDIX C

(Clause 4.4)

DETERMINATION OF DISPERSIBILITY

C-0. GENERAL

C-0.1 For the determination of dispersion of titanium dioxide, high speed shear mill (see C-1.1) is used. It consists of a stainless steel cup with a Cowles type impeller. Effective use of high speed disperser depends on establishing a satisfactory relationship between all the variables, the most important of which are the composition and volume of the mill base, dimensions of the impeller and the tank, location of the impeller within the tank and impeller speed. Some of these are inter-dependent and balance shall be worked out for each individual system.

C-0.2 Mill Base Formulation — For good results, a high mill base viscosity is essential, but alone it is not enough. By using a medium of higher solids content, the pigment particles are not wetted properly and hence for better results the medium solids are kept as low as possible. This facilitates wetting and the required adhesion at the impeller mill base interface. The limits for medium solids are 15 to 40 percent and when this medium is used to achieve high viscosity, the pigmentation has to be increased. For good results, the pigment content shall be 42 percent by volume calculated on the total volume of the mill base (approximately equal to 75 percent by mass). Even while doing accurate formulation for mill base, there are factors like dilatancy and thixotropy, which keep on changing, depending on the shear rate operated in the tank. The shear transmission problems can be usually overcome by modifying the mill base conditions or mill base composition. The following are some of the measures that may be employed.

a) Decrease the mill speed (this remedies only excessive dilatancy);
b) Use of narrow container (alternatively use a larger impeller and decrease the mill speed proportionately);
c) Increase the binder solids (40 percent should be regarded as maximum);
d) Change the solvent composition,
e) Change or modify the dispersion medium, namely, mill in an alkyd/amino mixture instead of alkyd resin alone; and
f) Decrease slightly the pigment content of the mill base.
C-1. APPARATUS

C-1.1 The dimensions for tank impeller and charge and the rpm shall be as follows

When \( D \) is the diameter of impeller, the tank shall have 2 to 3 \( D \) as internal diameter. The impeller shall be fixed at 0.5 \( D \) above the base of the tank. The charge to be added for better results shall have a depth of 1.8 \( D \). The impeller will have 1 050 to 1 350 metres per minute as peripheral speed.

Rotor speed, \( N = \frac{\text{Peripheral speed}}{D} \times 31.8 \)

C-2. MILLING TIME

C-2.1 10 to 15 Minutes — For satisfactory dispersion, a milling time of 10 minutes is recommended. Dispersion is said to be satisfactory if the required fineness is achieved in 10 minutes. If satisfactory dispersion is not induced within 15 minutes, still longer milling is most unlikely to improve the fineness.

C-3. PROCEDURE

C-3.1 Weigh the calculated amount of medium at 35 percent of non-volatile content (NVC) and the pigment in the container. Mix them using a spatula and transfer the contents to the tank. Run the impeller at the required speed. After 10 minutes check the fineness on a Hegman gauge.

C-3.2 Continue milling for another 5 minutes and check the fineness on Hegman gauge.

C-4. OBSERVATION

C-4.1 Compare the fineness reading of the sample with that of the approved sample.

APPENDIX D

( Clause 4.5 )

TEST FOR DURABILITY

D-0. GENERAL

D-0.1 The paint formulated as prescribed in E-1.2, E-1.3 and E-1.4, when tested in accelerated weathering unit of carbon arc type for 21 days...
as prescribed in A-4 of IS 2932-1974*, shall not show chalk resistance, gloss retention, colour retention, checking and cracking inferior to that of the approved sample. Xenon arc type weatherometer may also be used in place of carbon arc type weathering unit.

D-1. PROCEDURE

D-1.1 Test the paint formulated with the material as given in D-0 by the accelerated weathering method prescribed in A-4 of IS 2932-1974*. Evaluate the performance of the material by the method of rating prescribed in Appendix A of IS 2932-1974*. Treat the approved sample in a similar manner and subject to accelerated weathering tests simultaneously. The material shall be taken to have passed the requirement for durability if the performance of the paint prepared with the sample under test is not inferior to that of the paint prepared with the approved sample in respect of characteristics for gloss retention, chalk resistance, colour retention, checking and cracking.

APPENDIX E

[Table 1, Item (ii)]

DETERMINATION OF MASS COLOUR

E-0. GENERAL

E-0.1 For the determination of mass colour, paints of sample under test and the approved sample are prepared. The quantification of colour values may be made by the use of instrument, wherever possible; otherwise a drawdown may be drawn of the sample under test and the approved sample and visually compared.

E-1. PAINT PREPARATION

E-1.1 Requirements

E-1.1.1 Alkyd Resin Medium — Long semi-drying oil, alkyd, with pentaerythritol with oil length of 60 to 65 percent

E-1.1.2 Pigments — 30 percent by mass.

E-1.1.3 Pigment-Volume-Concentration (PVC) — 20 percent.

*Specification for enamel, synthetic, exterior a) undercoating, b) finishing (first revision)*
E-1.1.4 **Solvents** — Petroleum hydrocarbon solvents (*see IS : 1745-1978*).

E-1.1.5 **Driers** — The following driers on metal content basis of the non-volatile matter in the medium shall be used:
   a) 0.06 percent cobalt octoate, and
   b) 0.5 percent lead octoate.

E-1.1.6 **Film Application** — with applicator of 50 microns so as to give dry film thickness of approximately 25 microns.

E-1.2 **Formulation for Air-Drying Alkyd Gloss Paint**

E-1.2.1 The paint shall be prepared as follows.

- E-1.2.1.1 In 220 ml, glass jar take the following materials:
  a) 130 g of gloss ballotini,
  b) 60 g of titanium dioxide (TiO₂), and
  c) 20 g of alkyd resin at 20 percent non-volatile component (NVC).

- E-1.2.1.2 Ball mill for 18 ± 2 hours.

- E-1.2.1.3 Then add:
  a) 10 ml of petroleum hydrocarbon solvent, low aromatic (*see IS 1745-1978*),
  b) 7.5 ml of mixed driers,
  c) 0.5 ml of methyl ethyl keto (MEK) oxime or equivalent antiskinning agent, and
  d) 101 g of alkyd resin at 70 percent NVC.

- **OR**

- E-1.2.1.4 In 1,000 ml glass jar add:
  a) 600 g of glass ballotini,
  b) 300 g of TiO₂,
  c) 100 g of alkyd resin at 20 percent NVC.

- E-1.2.1.5 Ball mill for 18 ± 2 hours.

*Specification for petroleum hydrocarbon solvents (*second revision*)
Then add

a) 50 ml of petroleum hydrocarbon solvent, low aroma (see IS : 1745-1978*),
b) 30 ml of mixed driers,
c) 2 ml of methyl ethyl keto (MLK) oxime or equivalent anti-skinning agent, and
d) 500 g of alkyd resin at 70 percent NVC.

Into a clean jar, weigh appropriate amounts of ballotini, resin solution and the pigment under test as described above. In another clean jar, weigh the identical quantities of the approved pigment. Screw the lids on to the jars and shake till the pigment is wetted.

Place the jars on a suitable milling equipment (trundler) and mill its contents overnight (18 ± 2 h).

Scrap any dry pigment still adhering to the neck of the jar or to the lid after 30 minutes trundling into the mill base.

After the milling, remove the jar from the trundler. Check the finish of the mill base on Heginan gauge, this should be less than 10 microns. Then add the medium, solvent, driers and antiskinning agent. Add the calculated quantity, so that the paint shall have 20 percent pigment volume concentration. Return the jars on trundlers and mix the mill base thoroughly. Remove the ballotini by pouring the contents of the jar through a metal filter funnel. Collect the filtrate in a suitable can.

**E-1.3 Formulation for Exterior Emulsion Paint**

**E-1.3.1 Pigment : Binder Ratio — 1.86 : 1.**

**E-1.3.2 Milling Vessel — 11 cm high and 8.5 cm in diameter, laboratory high speed impeller mill (Cowles type impeller).**

**E-1.3.3 Materials —** Add the following ingredients in the milling vessel as specified:

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>Content</th>
</tr>
</thead>
<tbody>
<tr>
<td>TiO₂</td>
<td>212 g</td>
</tr>
<tr>
<td>Talc</td>
<td>79 g</td>
</tr>
<tr>
<td>Barytes</td>
<td>44 g</td>
</tr>
</tbody>
</table>

*Specification for petroleum hydrocarbon solvents (second revision).*
E-1.3.4 Mill for 5 minutes at 12 000 rpm.

E-1.3.5 In one litre of the material weigh as given below

a) Vinyl acetate copolymer (56 percent non-volatile); and
b) Mill base 440 g

E-1.3.6 Stir by hand for 10 minutes for even and homogeneous mixing.

NOTE — A specific percentage solution of HEC which will have viscosity of 50 to 60 p on Brookfield, RVT model on spindle No 6, 50 rev/min

E-1.4 Formulation of Stoving Alkyd Gloss Paint

E-1.4.1 Ingredients

E-1.4.1.1 Alkyd resin — Short coconut oil alkyd with glycerol with oil length of 30 to 40 percent.

E-1.4.1.2 Pigment — 18 percent by mass

E-1.4.1.3 Pigment volume concentration (PVC) — 10 percent.

E-1.4.1.4 Pigment and binder ratio — 1:2

E-1.4.1.5 Alkyd and malamine formaldehyde ratio — 4:1

E-1.4.1.6 Solvents — Xylene and butyl alcohol.

E-1.4.1.7 Additives — Zinc naphthenate having metal content of 2 percent solution in petroleum hydrocarbon solvents.

E-1.4.2 Paint Preparation

E-1.4.2.1 In 2 000 ml, porcelain pot mill, take the following materials:

a) 1 200 ml of pebbles;
IS : 9788 - 1981

b) 540 g of titanium dioxide;
c) 170 g of alkyd resin at 40 percent non-volatile component, and
d) 15 g of zinc naphthenate solution.

Ball mill for 36 h to less than 5 microns, then add the following.
i) 60 g of xylene,
ii) 120 g of butyl alcohol;
iii) 1 640 g of alkyd resin at 50 percent non-volatile component,
iv) 400 g of malamine formaldehyde resin at 55 percent non-volatile component, and
v) 5 g of xylene to adjust viscosity

E-1.5 Preparation and Examination of Panels

E-1.5.1 Place a white card or some other preferred substrate on a convenient horizontal surface.

E-1.5.2 Transfer the paint under test to the card giving a pool that is adjacent to but separated from the pool of similarly made paint containing a standard pigment. Draw down the paints with a smooth stroke of a wire wound applicator. Place the panel horizontally in a ventilated drying cabinet and allow the paint to dry overnight.

E-1.5.3 Visually compare the dry paint film with the standard paint film under approved artificial lightening conditions. A numerical assessment of colour may be made, provided a suitable colourmeter is available

APPENDIX F

[ Table 1, Item (iii) ]

DETERMINATION OF REDUCING POWER

F-0. GENERAL

F-0.1 Titanium dioxide is tinted with diluted phthalocyanine green pigment and the reducing power is determined. The reducing power is expressed in the same manner as in Reynold Number. In Reynold Number, ultramarine blue pigment is used; here it is replaced by phthalocyanine green and the tinting strength of the pigment is being designated as 'ISI Tinting Strength Number'.
F-1. PROCEDURE

F-1.1 Diluted Phthalocyanine Green Powder — Dry phthalocyanine green pigment and blane fixe separately at 105°C for 2 h or till free of moisture. Take 1 g of dried powder of phthalocyanine green and 99 g of dried powder of blane fixe in a suitable porcelain jar containing 20 mm porcelain heavy density balls. Grind the mixture for 24 h and then collect and store in a well stoppered bottle. Use the mixture for determination of ISI Tinting Strength Number.

F-1.2 Weigh 0.5 g of the titanium dioxide pigment under test and phthalocyanine green powder as per the chart given in Table 2. Mix on mullet with acid refined linseed oil conforming to IS 75-1973* and prepare a paste. Draw a drawdown of sample and standard on a glass plate using a suitable applicator.

<table>
<thead>
<tr>
<th>SL NO</th>
<th>ISI TINTING STRENGTH NO</th>
<th>PHTHALOCYANINE GREEN POWDER IN g</th>
<th>OIL IN ml</th>
</tr>
</thead>
<tbody>
<tr>
<td>i)</td>
<td>1 500</td>
<td>1.500</td>
<td>0.58</td>
</tr>
<tr>
<td>ii)</td>
<td>1 600</td>
<td>1.600</td>
<td>0.61</td>
</tr>
<tr>
<td>iii)</td>
<td>1 700</td>
<td>1.700</td>
<td>0.64</td>
</tr>
<tr>
<td>iv)</td>
<td>1 800</td>
<td>1.800</td>
<td>0.67</td>
</tr>
</tbody>
</table>

F-1.3 Observation — Observe the reducing power immediately and record the result.

APPENDIX G

[ Table 1, Item (ix) ]

DETERMINATION OF RESISTIVITY OF AQUEOUS EXTRACT

G-0. GENERAL

G-0.1 The resistivity (inverse of conductivity) of the aqueous extract is determined to know the ionic components, if present in the titanium dioxide. The titanium dioxide pigment used in surface coating has applications like electrostatic spraying and electrodeposition wherein the resistivity is of prime importance.

*Specification for linseed oil, raw and refined (second revision)
IS : 9788 - 1981

G-1. MATERIALS REQUIRED

G-1.1 Conductivity Bridge

G-1.2 Standard Conductivity Cell

G-1.3 Conductivity Water — Distil redistilled water with a pinch of potassium permanganate till it achieves a resistivity of 1 00 000 Ω cm minimum.

G-1.4 Erlenmeyer Flask, Funnel and Centrifuge Tube — 30 cm in diameter.

G-2. PROCEDURE

G-2.1 Take 10 g of the sample under test in Erlenmeyer flask previously boiled in and washed with distilled water. Add 93 ml of conductivity water and weigh the flask along with the contents. Boil the suspension for 10 minutes over a moderate flame keeping a funnel in the mouth of the flask. Allow the contents to cool to room temperature. Wash the sides of the flask and funnel stem with conductivity water and collect the filtrate in the same flask. Weigh the flask and add requisite quantity of conductivity water such that the total mass of pigment and water is 100 g.

G-2.2 Swirl the flask and divide the contents into two equal parts and transfer to centrifuge tubes. Centrifuge the suspension at 5 000 rpm for about 30 minutes and decant the clear solution to a clean Erlenmeyer flask. Wash the conductivity cell several times with conductivity water and finally with a portion of the solution whose conductance is to be determined. Suspend the conductivity cell in the solution and set the appropriate range of resistance on the instrument. Turn the pointer on the dial to such a position till the indicator shows a null balance. Read the value of resistance.

G-2.3 Repeat the procedure three times for accurate results. Determine the resistance of the standard sample in identical manner.

G-3. CALCULATIONS

G-3.1 Determine the resistivity by dividing the average of the three resistance readings by the cell constant C.

\[
\text{ Resistivity } = \frac{1}{X} = \frac{R}{C} \text{ and expressed in } \Omega \text{ at a specified temperature }
\]
where

\[ X = \text{conductivity}, \]
\[ R = \text{determined resistance}, \text{ and} \]
\[ C = \text{cell constant}. \]

For the cell constant measure the resistance of 0.02 \( M \) and 0.01 \( M \) solutions of potassium chloride in conductivity water. These solutions have resistivities 417 and 816 \( \Omega \) at 18°C respectively.

\[
\text{Cell constant } (C) = \frac{\text{Determined resistance}}{\text{Resistance of potassium chloride solution}}
\]

**APPENDIX H**

[Table 1, Item (x)]

**DETERMINATION OF WATER DEMAND**

**H-0. GENERAL**

H-0.1 The water demand test is carried out to know the surface area in general and the type of surface treatments. It also reveals the particle size and the extent of grinding, particularly for rutile of \( \text{R}_3 \) grade. It facilitates formulation of mill base composition.

**H-1. MATERIALS REQUIRED**

H-1.1 Stainless Steel Cup

H-1.2 Stainless Steel Spatula

H-1.3 Burette — 0.1 ml graduation.

H-1.4 Sodium Hexametaphosphate — 5 percent solution.

**H-2. PROCEDURE**

H-2.1 Take about 50 g of titanium dioxide accurately weighed in the steel cup and add 0.4 percent equivalent on mass basis of pigment, sodium hexametaphosphate solution. Add from the burette drop by drop distilled water to the powder and mix the powder well. After the paste is formed add water very carefully and slowly till the flow point is achieved. Note the reading on the burette. Calculate the total water by adding the burette reading and volume in ml of sodium hexametaphosphate solution.
H-3. CALCULATION

H-3.1 Express the result as ml of water required for 100 g of pigment

NOTE — Sodium hexametaphosphate is used so that the aggregate and agglomerates are broken down and individual particles are wetted

APPENDIX J

[Table 1, Item (xi)]

SETTLING TEST

J-0. GENERAL

J-0.1 Settling tests for air drying and stoving paints are prescribed.

J-1. OUTLINES OF THE METHOD

J-1.1 Test for settling properties of an drying and stoving paints prepared with known amount of titanium dioxide pigment and alkyd resin of specified oil length is conducted and observations recorded.

J-2. TEST ON AIR DRYING PAINT

J-2.1 Formulation of Paint — Prepare an air drying paint with drying/semi-drying oil alkyd having 60 to 62 percent oil length, 24 to 26 percent titanium dioxide and 60 percent solids in petroleum hydrocarbon solvent. The pigment to binder ratio shall be 1 : 1.3. Disperse all these ingredients in a ball or sand mill for Hegman gauge fineness of 0 mil. The viscosity of this paint shall be 100 to 120 seconds at 30°C in IS cup No. 4

J-2.2 Procedure — Thin to 30 seconds viscosity at 30°C in IS cup No. 1 with petroleum hydrocarbon solvent, low aromatic (see IS 1745-1978*). Fill it in a 500 ml container to within 1 cm of the top. Close the container and keep for 72 h at room temperature.

J-2.3 Observation — Open the container after 72 h and observe the condition of the material. The requirement of this test shall be taken to be satisfied if there is no settling, sedimentation or separation of the pigment.

*Specification for petroleum hydrocarbon solvents (second revision).
J-3. TEST ON STOVING PAINT

J-3.1 Formulation of the Paint — Prepare a stoving paint with coconut oil alkyd having 40 percent oil length, 34 percent titanium dioxide content and 60 percent solids in xylene. The pigment to binder ratio shall be 1.2 and alkyd to melamine formaldehyde ratio 3.5 1. Disperse the ingredients in ball or sand mill for Hegman gauge fineness of 0 mil. The viscosity of this paint shall be 60 seconds in IS cup No. 4 at 30°C.

J-3.2 Procedure — Thin to 18 to 20 seconds viscosity at 30°C in IS cup No. 4 with xylene and butanol in ratio of 9:1. Fill it in a 500 ml container to within 1 cm of the top. Close the container and keep it for 72 h at room temperature.

J-3.3 Observation — Open the container after 72 h and observe the condition of the material. The requirement of this test shall be taken to be satisfied if there is no settling, sedimentation or separation of the pigment.
Members Representing

SHRI S BHATTACHARYYA
Alkali & Chemical Corporation of India Ltd, Calcutta

SHRI M. L. MALHOTRA (Alternate)

SHRI T. K. S. MANI
Addisons Paints & Chemicals Ltd, Madras

SHRI V C PANCHAMIYA
Kamam Metallic Oxides Pvt Ltd, Bombay

SHRI K L RATHI
Sudarshan Chemical Industries Ltd, Pune

SHRI H J RATHI (Alternate)

DR V S VIJAYAN NAYAR
Travancore Titanium Products Ltd, Trivandrum

SHRI D V WITTENDARKER
Waldies Limited, Calcutta