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भारतीय मानक मसौदा

**रबर उद्योग के लिए टाइटेनियम डाइऑक्साइड (एनाटेज़ प्रकार) – विशिष्टता**  
(IS 8862 का पहला पुनरीक्षण)

*Draft Indian Standard*

**TITANIUM DIOXIDE (ANATASE TYPE) FOR RUBBER INDUSTRY —  
SPECIFICATION**

*(First Revision of IS 8862)*

ICS 71.060.20

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Rubber and Rubber Products Sectional  
Committee, PCD 13

Last date for receipt of comment is  
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**FOREWORD**

*(Formal clauses will be added later)*

This standard was originally published in 1978. Titanium dioxide, anatase, one of the two crystalline forms of titanium dioxide is used in the rubber industry as a white pigment for the manufacture of white and light coloured rubber products. The distinction between the two forms, namely rutile and anatase can be done by X-ray crystallography. The same was, however not been included in the specification as the facilities for doing this test are not widely available.

This revision has been undertaken to update the cross-referred standards in the standard and editorial changes.

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.

## 1 SCOPE

This standard prescribes the requirements and methods of sampling and test for titanium dioxide (anatase type) in powder form intended for use as a white pigment in rubber compounds.

## 2 REFERENCES

The following standards contain provisions which, through reference in this text, constitute provisions of the standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on the standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<i>IS No.</i>	<i>Title</i>
IS 264 : 2005	Nitric acid — Specification ( <i>third revision</i> )
IS 265 : 2021	Hydrochloric acid — Specification ( <i>fifth revision</i> )
IS 266 : 1993	Sulphuric acid — Specification ( <i>third revision</i> )
IS 7086 (Part 1) : 1973	Methods of sampling and test for rubber compounding ingredients: Part 1

## 3. REQUIREMENTS

**3.1 Appearance** — The material shall be in the form of a white fine powder, free from lumps, grit and extraneous matter.

**3.2** The material shall comply with the requirements given in Table 1 when tested according to the methods listed in col (4) of the table.

**Table 1 Requirements of Titanium Dioxide (Anatase Type) For Rubber Industry**  
(Clauses 3.2, 5.2 and 5.3)

<b>Sl No.</b>	<b>Characteristic</b>	<b>Requirement</b>	<b>Method of Test, Ref To Clause of IS 7086 (Part 1) and Annex</b>
(1)	(2)	(3)	(4)
i)	Colour	Close match to the approved sample	Visual Examination
ii)	Sieve residue, percent by mass, <i>Max</i> : a) Through 150 micron IS Sieve b) Through 45 micron IS Sieve	0.01 0.05	3 of IS 7086 (Part 1)
iii)	Relative density at 27 °C/27 °C	3.85 to 3.95	4 of IS 7086 (Part 1)
iv)	pH	6 to 8	5 of IS 7086 (Part 1)

v)	Moisture content, percent by mass, <i>Max</i>	0.5	7 of IS 7086 (Part 1)
vi)	Matter soluble in water, percent by mass, <i>Max</i>	0.5	8 of IS 7086 (Part 1)
vii)	Titanium dioxide (as TiO <sub>2</sub> ), percent by mass, <i>Min</i>	98.0	Annex A
viii)	Copper, percent by mass, <i>Max</i>	0.002	Annex B
ix)	Iron, percent by mass, <i>Max</i>	0.04	Annex C
x)	Solubility in mixture of sulphuric acid and ammonium sulphate	To pass the test	Annex D

## **4 PACKING AND MARKING**

### **4.1 Packing**

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

### **4.2 Marking**

Each package shall be securely closed and legibly marked to furnish the following information:

- a) Manufacturer's name and registered trade-mark, if any;
- b) Name of the material;
- c) Net mass of the material in the package; and
- d) Lot, batch number and year of manufacture.

#### **4.2.1 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 thereunder, and the products may be marked with the Standard Mark.

## **5 SAMPLING AND CRITERIA FOR CONFORMITY**

### **5.1 Sampling**

The sampling shall be done as prescribed in **15** of IS 7086 (Part 1).

### **5.2 Number of Tests**

Tests for all the characteristics (*see* Table 1) shall be conducted on a composite sample.

### **5.3 Criteria for Conformity**

The lot shall be considered as conforming to the specification, if the composite sample satisfies all the requirements given in Table 1.

**ANNEX A**  
[Table 1, Sl no. (vii)]  
**DETERMINATION OF TITANIUM DIOXIDE**

**A-1 GENERAL**

**A-1.1** Two methods for the 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 applicable.

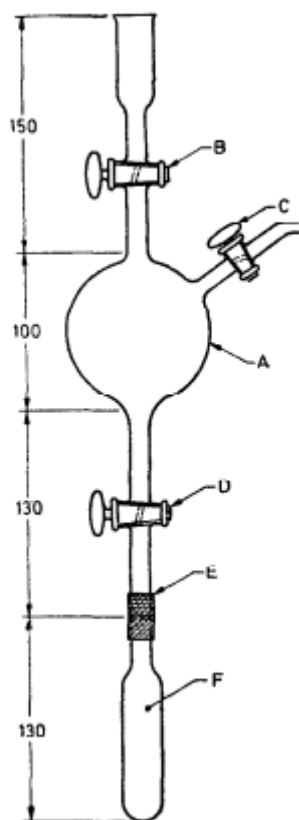
**A-2 METHOD 1 — ZINC REDUCTION METHOD**

**A-2.1 Outline of the 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 trivalent titanium is titrated with ferric alum.

**A-2.2 Apparatus**

**A-2.2.1 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.



All dimensions in millimetres  
**FIG.1 NAKAZONO REDUCTOR**

### **A-2.3 Reagents**

**A-2.3.1 Concentrated Sulphuric acid**, relative density 1.84 (see IS 266)

**A-2.3.2 Ammonium Sulphate**

**A-2.3.3 Zinc Amalgam** (3 percent)

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 about 25 g of zinc in small granules. Stir from time to time and replenish the dilute acid with water as required. When all the zinc has disappeared, allow the amalgam to cool and stand for several hours. Finally filter through a Gooch crucible with no asbestos pad. Store the amalgam in glass bottle under 2 percent (*m/v*) sulphuric acid.

**A-2.3.4 Standard Ferric Alum Solution** (0.062 5 N)

Transfer by a pipette 50 ml of the ferric alum solution to be standardized to the Nakazono reductor prepared ready for use as described in **A-2.4.2**. Displace the air in the reductor by passing carbon dioxide for 3 min. Close the taps *B* and *C*. Bring the amalgam into intimate contact with ferric alum solution by shaking the apparatus vigorously for 5 min. Place the apparatus on a ring stand. Drain the amalgam and pass inert gas as described under **A-2.4.4**. Titrate it against standard potassium permanganate. The end point is the appearance of light pink colour at least persistent for 30 s. Calculate the strength of the ferric alum solution as follows:

$$\text{Strength of ferric alum solution} = V \times \frac{0.1}{50}$$

where

*V* = volume of 0.1 N standard potassium permanganate solution required for titration.

**A-2.3.5 Potassium Thiocyanate Solution**, 10 percent (*m/v*)

### **A-2.4 Procedure**

**A-2.4.1** 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 then over a strong flame until solution is complete, observing caution while examining the solution. Cool the solution, dilute with 100 ml of distilled water, stir and filter, if necessary.

**A-2.4.2** Open stopcock *C*, transfer sufficient quantity of 4 percent sulphuric acid to fill the bottom flask and leave no air space below the stopcock *D*. Close tap *D* and add 20 ml of zinc amalgam to *A*. The reductor is now ready for use.

**A-2.4.3** 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 min. 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 min., holding it in

such a way that the stopcocks are kept shut and in position. The rate of reduction is apparent by development of the characteristic violet colour of the trivalent titanium.

**A-2.4.4** Place the apparatus on a ring stand. Open stopcock *D* slightly and allow the amalgam to flow slowly into *F*. 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 potassium thiocyanate solution and titrate with standard ferric alum solution until a faint pink coloration persists for one minute.

**A-2.4.5** 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-2.5 Calculation**

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

where

*V* = volume in ml of 0.062 5 N ferric alum solution required, and

*M* = mass in g of pigment taken

## **A-3 METHOD 2 — ALUMINIUM REDUCTION METHOD**

### **A-3.1 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-3.2 Apparatus**

#### **A-3.2.1 Delivery Tube**

Made of about 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-3.2.2 Long Necked Flask, 500 ml capacity**

### **A-3.3 Reagents**

#### **A-3.3.1 Ammonium Sulphate**

#### **A-3.3.2 Sulphuric acid, relative density 1.84 (see IS 266)**

#### **A-3.3.3 Hydrochloric acid, relative density 1.19 (see IS 265)**

#### **A-3.3.4 Aluminium Foil, electrolytic grade**

#### **A-3.3.5 Sodium Bicarbonate Solution, saturated**

#### **A-3.3.6 Ammonium Thiocyanate Indicator Solution**

Dissolve 24.5 g of ammonium thiocyanate in 80 ml of hot water. Filter, cool to room temperature, dilute to 100 ml and preserve in well stoppered, amber coloured bottle.

### **A-3.3.7 Standard Ferric Ammonium Sulphate Solution**

Dissolve 30.16 g of fresh ferric ammonium sulphate [ $\text{FeNH}_4(\text{SO}_4)_2 \cdot 12 \text{H}_2\text{O}$ ] in 800 ml of water containing 15 ml of concentrated sulphuric acid. Add 0.1 N potassium permanganate solution until a very slight pink colour is obtained. Dilute to 1000 ml and mix thoroughly. Filter, if necessary. Standardize with 0.190 g to 0.210 g of standard titanium dioxide of known purity prescribed in **A-3.4** and calculate titanium dioxide equivalent of solution in grams of titanium dioxide per millilitre of solution as given below:

$$\text{Titanium dioxide equivalent} = \frac{M \times P}{V \times 100}$$

where

$M$  = mass in g of standard titanium dioxide taken for test,

$P$  = percentage of titanium dioxide in standard titanium dioxide used, and

$V$  = volume in ml of ferric ammonium sulphate solution required for titration.

### **A-3.4 Procedure**

**A-3.4.1** Weigh accurately about 0.190 g to 0.210 g of the dry material and transfer the same to a 500 ml long necked flask. Add 7 g to 9 g of ammonium sulphate and 20 ml of sulphuric acid, mix carefully, heat on a hot plate to fuming and then over a strong flame until solution is complete. Cool and add 120 ml of water and 20 ml of hydrochloric acid. Boil the contents and remove from heat.

**A-3.4.2** 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 nearly 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 at the same time that the long end of the delivery tube is near the bottom of a 250 ml beaker containing about 150 ml of sodium bicarbonate solution.

**A-3.4.3** Heat the flask as soon as the dissolution of aluminium is complete to gentle boiling for 3 min to 5 min 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 rod attached to it with a little water catching the rinsing in the flask. Add 2 ml of ammonium thiocyanate indicator solution and titrate immediately against standard ferric ammonium sulphate solution to a straw coloured end point.

### **A-3.5 Calculation**

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

where

$V$  = volume in ml of ferric ammonium sulphate solution used in test,

$E$  = titanium dioxide equivalent (*see A-3.3.7*) of ferric ammonium sulphate solution in g/ml, and

$M$  = mass in g of material taken for test.

**ANNEX B**  
[Table 1, Sl. no. (viii)]  
**DETERMINATION OF COPPER**

**B-1 APPARATUS**

**B-1.1 Beaker**

**B-1.2 Nessler cylinder**

**B-1.3 Burette**

**B-2 REAGENTS**

**B-2.1 Standard Copper Sulphate Solution**

Weigh 0.393 g of copper sulphate ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ) into a small beaker and dissolve in water. Cautiously add 5 ml of concentrated sulphuric acid (relative density 1.84) and transfer to a 1000 ml volumetric flask. Dilute to the mark and shake to ensure homogeneity. Pipette 10 ml of this solution into a 100 ml volumetric flask, dilute to the mark with water and mix. Each millilitre of the final solution contains 0.000 01 g copper (as Cu).

**B-2.2 Concentrated Hydrochloric Acid**, relative density 1.19 (*see IS 265*)

**B-2.3 Nitric Acid**, relative density 1.40 (*see IS 264*)

**B-2.4 Citric Acid Solution**, Dissolve 10 g of citric acid in 100 ml of water

**B-2.5 Ammonium Hydroxide Solution**

**B-2.6 Sodium Diethyldithiocarbamate**

Moisten about 0.01 g of gum tragacanth with about 1 ml of acetone and pour into 1 litre of boiling water. When cool, dissolve in this solution 1 g of sodium diethyldithiocarbamate.

**B-3 PROCEDURE**

**B-3.1** Weigh 5 g of the sample, accurately weighed, into a 200 ml beaker, add 200 ml of concentrated hydrochloric acid and 4 ml of concentrated nitric acid. Cover with glass and heat for one hour with occasional stirring. Dilute to 40 ml with water and add 10 ml of citric acid solution followed by ammonium hydroxide, added dropwise, until the solution is just alkaline to litmus and then 2 ml in excess. Bring to boil and allow to stand for 15 min to 20 min in warm place. Filter into a 100 ml Nessler cylinder, wash with distilled water until the volume of the filtrate is 70 ml to 80 ml. Add 10 ml of sodium diethyldithiocarbamate solution and make up to 100 ml with distilled water.



**B-3.2** Prepare a blank solution from the same quantities of reagents diluted to 100 ml in a second Nessler cylinder.

**B-3.3** By means of a burette add standard copper sulphate solution to the blank, and equal volumes of water to the sample solution until the colour intensities in the two cylinders are identical. Note the volume of copper solution required.

#### **B-4 CALCULATION**

**B-4.1** Calculate the copper content as follows:

$$\text{Copper content, percent by mass} = \frac{V \times 0.001}{M}$$

where

$V$  = volume in ml of standard copper solution required, and  
 $M$  = mass in g of the sample taken.

**ANNEX C**  
[Table 1, *Sl No.* (ix)]  
**DETERMINATION OF IRON**

#### **C-1 APPARATUS**

**C-1.1 Beaker**

**C-1.2 Nessler cylinder**

**C-1.3 Burette**

#### **C-2 REAGENTS**

**C-2.1 Dilute Hydrochloric acid**, approximately 0.2 N

**C-2.2 Concentrated Hydrochloric acid**, relative density 1.19 (*see* IS 265)

**C-2.3 Ammonium Persulphate**, solid

**C-2.4 Butanolic Potassium Thiocyanate**

Dissolve about 10 g of potassium thiocyanate in 10 ml of water at  $(27 \pm 2)$  °C. Add n-butyl alcohol to make up to 100 ml and shake vigorously until the solution is clear.

**C-2.5 Dilute Sulphuric Acid**, 10 percent (v/v)

**C-2.6 Standard Iron Solution**

Dissolve 0.702 g of ammonium ferrous sulphate  $[(\text{NH}_4)_2\text{SO}_4 \cdot \text{FeSO}_4 \cdot 6\text{H}_2\text{O}]$  in 20 ml of water containing 5 ml of concentrated sulphuric acid and dilute with water to 1000 ml. One millilitre of the solution contains 0.1 mg of iron (as Fe).

### **C-3 PROCEDURE**

**C-3.1** Take approximately 5 g of moisture free sample of titanium dioxide weighed accurately to 0.001 g in a 400 ml beaker and boil with 20 ml of dilute hydrochloric acid, till a little acid (approximately 5 ml) is left. Further add 15 ml of concentrated hydrochloric acid and slowly boil till some acid is left as before. Add 20 ml of distilled water and boil for 5 min. Filter hot through a filter paper (Whatman No. 42 or equivalent) collecting the filtrate in a 400 ml beaker. If the filtrate appears turbid, filter again through the same filter paper, collecting the filtrate in another 400 ml beaker. Wash the residue in filter paper several times with hot water, till a drop of washing is neutral to blue litmus paper. Evaporate the total filtrate to less than 75 ml, cool to room temperature and transfer to a 100 ml volumetric flask. Wash the beaker and transfer the washings to 100 ml flask and make up the volume.

**C-3.2** Take 20 ml of the solution from the volumetric flask in a Nessler cylinder. Add 30 mg of ammonium persulphate and 15 ml of butanolic potassium thiocyanate and make up to 50 ml. Shake vigorously for 30 s and allow the liquids to separate. Carry out a control test in the second Nessler cylinder using 4 ml of standard iron solution and the same quantities of other reagents in the same volume of the reaction mixture. Match the colour produced in the butanol layers after 2 min.

**C-3.3** The limit prescribed shall be taken to have not been exceeded if the intensity of colour with the sample is not greater than that in the control test.

## **ANNEX D**

*[Table 1, Sl No. (x)]*

### **SOLUBILITY IN MIXTURE OF SULPHURIC ACID AND AMMONIUM SULPHATE**

#### **D-1 PROCEDURE**

**D-1.1** Dissolve 0.2 g of the titanium dioxide in a mixture of 25 ml of concentrated sulphuric acid and 10 g of ammonium sulphate. Add a few drops of hydrogen peroxide. Deep orange colour should develop.