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BUREAU OF INDIAN STANDARDS

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Draft Indian Standard

RED OXIDE OF IRON FOR RUBBER INDUSTRY — SPECIFICATION

(*Third Revision of IS* 1684)

ICS 71.060.20

Rubber and Rubber Products Sectional Committee, PCD 13	Last date for receipt of comment is **17 May 2024**

FOREWORD

(*Formal clauses will be added later*)

This standard was first revised in 1973 and further revised in 1994.

In the first revision, the requirements for colour and matter insoluble in hydrochloric acid were added and requirement for siliceous matter was deleted.

In the second revision, two types of red oxides namely natural and synthetic was specified; venetian red has, however, been excluded.

Third revision of this standard has been undertaken to incorporate various editorial corrections, updation of references to ensure accuracy and relevance in the revised standard.

For the purpose of deciding whether a particular requirement of their 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

1.1 This standard prescribes the requirements and methods of sampling and tests for natural and synthetic red oxides of iron intended for use in the rubber industry.

1.2 This standard does not cover the requirements for Venetian red.

2 REFERENCES

The following standards contains provision which, through reference in this text, constitute provisions of this standard. At the time of publication the edition 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 standard indicated below:

IS No.	*Title*
IS 33 : 1992	Methods of sampling and test for inorganic pigments and extenders for paints (*third revision*)
IS 265 : 2021	Hydrochloric acid — Specification (*fifth revision)*
IS 266 : 1993	Sulphuric acid — Specification (*third revision)*
IS 1070 : 2023	Reagent grade water — Specification (*fourth revision)*
IS 7086 (Part 1) : 1973	Methods of sampling and test for rubber compounding ingredients (Part 1)

3 TYPES

3.1 Type A — Natural red oxides of iron for rubber Industry

3.2 Type B — Synthetic Red oxide of iron for rubber industry (excluding Venetian red)

4 REQUIREMENTS

4.1 Description

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

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

5 PACKING AND MARKING

5.1 Packing

The Packing of the material shall be done as agreed to between the purchaser and the supplier.

5.2 Marking

5.2.1 Each package shall be marked with the following:

a) Name of the material;
b) Indication of the source of manufacture;
c) Net mass of the material;
d) Month and year of manufacture; and
e) Lot or batch number.

5.2.2 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act,* 2016 and the rules and regulations framed thereunder, and the products may be marked with the Standard Mark.

6 SAMPLING

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

6.2 Number of Test

6.2.1 Tests for manganese and copper (*see* Table 1) shall be conducted on individual samples.

6.2.2 Tests for all other characteristics shall be conducted on a composite sample.

6.3 Criteria for Conformity

6.3.1 *For Individual Samples*

6.3.1.1 *Copper*

Each individual test result shall satisfy the requirement of the specification as given in Table 1.

Table 1 Requirements of Red Oxides of Iron for Rubber Industry
(Clauses 4, 6.2.1, 6.3.1.1 *and* 7.1)

Sl No.	**Characteristic**	**Requirement**		**Methods of Test, Ref to**	
		Type A	Type B	Annex	Cl. No. in IS
(1)	(2)	(3)	(4)	(5)	(6)

i)	Colour	To match the approved sample		-	9 of IS 33
ii)	Staining power and tone	-	-do-	-	10 of IS 33
iii)	Sieve residue, percent by mass, *Max* a) through 75 micron IS Sieve b) through 150-micron IS Sieve	 0.40 0.01	 - 0.01	 - -	3 of IS 7086 (Part 4)
iv)	Relative density 27/27°C	5.0 to 5.5	5.0 to 5.5	- -	4 of IS 7086 (Part 1)
v)	Acidity (as H_2SO_4), percent by mass, *Max*	0.02	0.1	-	6 of IS 7086 (Part 1)
vi)	Volatile matter, percent by mass, *Max*	-	0.5	-	7 of IS 7086 (Part 1)
vii)	Moisture content, percent by mass, *Max*	0.5	-	-	7 of IS 7086 (Part 1)
viii)	Matter soluble in water, percent by mass, *Max*	2.0	0.5	-	8 of IS 7086 (Part 1)
ix)	Acid insoluble, percent by mass, *Max*	5.0	5.0	-	9 of IS 7086 (Part 1)
x)	Loss on ignition, percent by mass, *Max*	1.0	0.5	-	10 of IS 7086 (Part 1)
xi)	Manganese (as Mn), percent by mass, *Max*	0.05	0.05	-	11 of IS 7086 (Part 1)

xii)	Copper	To satisfy the requirement of test		-	12 of IS 7086 (Part 1)
xiii)	Iron oxide (as Fe_2O_3) percent by mass *Min*	90.0	95.0	A	-

6.3.1.2 *Manganese*

The mean and range of test results for manganese shall be calculated as follows:

$$\text{Mean } (\bar{X}) = \frac{\text{The sum of test results}}{\text{Number of test results}}$$

Range (*R)* = The difference between the maximum and the minimum value of the test results

The lot shall be deemed to have satisfied the requirements of the specification, *if* $\bar{X} + 0.6\,R \leq 0.05$.

6.3.2 *For composite sample*

In respect of all other characteristics, the lot shall be considered as conforming to the specification, if the composite sample satisfies each of these requirements.

7 TEST METHODS

7.1 Test shall be carried out according to the methods prescribed in Annex A and referred clauses of specification indicated in co1 6 of Table 1.

7.2 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
[*Table* 1, *Sl No.*(xiii) *and clause* 7.1]
DETERMINATION OF FERRIC OXIDE

A-1 GENERAL

Estimate ferric oxide volumetrically using a 0.1 N standard solution of potassium dichromate.

A-2 REAGENT

A-2.1 Concentrated Sulphuric Acid (*see* Conforming to IS 266)

A-2.2 Silica Gel

A-2.3 Concentrated Hydrochloric Acid (*see* Conforming to IS 265)

A-2.4 Stannous Chloride Solution

Dissolve 5 g of pure tin or 15 g of crystallized stannous chloride ($SnCl_2.2H_2O$) in about 50 ml of concentrated hydrochloric acid (relative density 1.16) and dilute with water to 200 ml.

A-2.5 Mercuric Chloride Solution

Dissolve 27 g of mercuric chloride in one litre of water.

A-2.6 Phosphoric Acid (Relative density 1.7)

A-2.7 Sulphuric Acid-Phosphoric Acid Mixture

Mix 15 ml of concentrated sulphuric acid and 15 ml of phosphoric acid and dilute to 100 ml. Keep this mixture as a ready stock.

A-2.8 Diphenylamine or Diphenyl Benzidine Indicator Solution

Dissolve one gram of diphenylamine or diphenyl benzidine in 100 ml of concentrated sulphuric acid.

A-2.9 Standard Potassium Dichromate Solution

Finely powder about 6 g of potassium dichromate (analytical reagent grade) in a glass or agate mortar, and heat for 30 min to 60 min in an air-oven at 140 °C to 150 °C. Cool in a desiccator. Dissolve about 4.9 g of the powder, accurately weighed, in distilled water in one litre measuring flask and shake thoroughly and make upto the litre mark.

$$\text{Normality of potassium dichromate solution} = \frac{M}{49.03}$$

where

M = mass of potassium dichromate dissolved in 1 000 ml of solution

A-3 PROCEDURE

A-3.1 Weigh accurately about 2 g of the material in a tared flat bottomed dish about 8 cm in diameter. Keep the dish with the material in an oven maintained at (105 $\pm$ 2) °C for two hours. At the end of this period, transfer the dish to a desiccator containing concentrated sulphuric

acid or silica gel and cool. Remove the dish and weigh. Repeat the heating and the weighing till constant mass is obtained.

A-3.2 Transfer about 0.3 g of the dried material into a beaker. Dissolve it in 25 ml of concentrated hydrochloric acid and warm gently to get the pigment (excepting siliceous matter) into complete solution. If dissolution of the material is difficult, digest the solution on hot-plate evaporating to dryness, and redissolve in concentrated hydrochloric acid as described above. Filter into an Erlenmeyer flask and make up the solution to about 50 ml. Add the stannous chloride solution, drop by drop until the solution just becomes colourless. Add two to three drops in excess, cool the solution and dilute to 150 ml to 200 ml with water. Add a slight excess of mercuric chloride solution, when a silky precipitate will be formed. Add 15 ml of sulphuric acid-phosphoric acid mixture. Add 3 drops (about 0.1 ml) of diphenylamine or diphenyl benzidine indicator, and titrate with standard potassium dichromate solution.

A-4 CALCULATION

$$\text{Ferric oxide, percent by mass} = \frac{7.98\ V\ N}{M}$$

where

V = volume in ml of standard potassium dichromate solution;

N = normality of standard potassium dichromate solution; and

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