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SPECIFICATION FOR NATURAL RED OXIDES OF IRON FOR RUBBER INDUSTRY

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0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 7 September 1960, after the draft finalized by the Rubber Products Sectional Committee had been approved by the Chemical Division Council.

0.2 Natural red oxide of iron is widely used in the rubber industry as a pigment. Rubber manufacturers since a long time have been experiencing difficulty in procuring suitable quality of raw materials for want of national standards. This standard has been formulated with a view to facilitating and assuring a supply of a standard quality of natural red oxides of iron to the rubber industry.

0.3 The Sectional Committee responsible for the preparation of this standard took into consideration the views of producers, consumers and technologists, and felt that it should be related to the prevailing trade practices in this field in the country. Due consideration has also been given to the quality of indigenous and imported natural red oxides of iron available to the industry.

0.4 This standard requires reference to the following Indian Standard Specifications of which the last one is a necessary adjunct to this standard:

IS: 265-1950 HYDROCHLORIC ACID
IS: 266-1950 SULPHURIC ACID
IS: 323-1959 RECTIFIED SPIRIT (*Revised*)
IS: 460-1953 TEST SIEVES
IS: 915-1958 ONE-MARK GRADUATED FLASKS
IS: 1070-1960 WATER, DISTILLED QUALITY (*Revised*)
IS: 1683-1960 BARYTES FOR RUBBER INDUSTRY

0.4.1 Wherever a reference to any Indian Standard mentioned in **0.4** or otherwise appears in this specification, it shall be taken as a reference to the latest version of the standard.

0.5 Natural red oxides of iron for paints have been covered in IS: 46-1950 Specification for Natural Red Oxides of Iron for Paints.

0.6 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: 2-1960 Rules for Rounding Off Numerical Values (*Revised*). The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

0.7 This standard is intended chiefly to cover the technical provisions relating to the material, and it does not include all the necessary provisions of a contract.

1. SCOPE

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

2. SAMPLING

2.1 Representative samples of the material shall be drawn as prescribed in Appendix A.

3. REQUIREMENTS

3.1 Description — The material shall be supplied in the form of dry powder.

3.2 Fineness — When tested according to the method prescribed in Appendix B of IS: 1683-1960, the material shall comply with the following requirements:

Retained on *IS Sieve 8 (aperture 75 microns), percent by weight, *Max*	0·4
Retained on *IS Sieve 15 (aperture 151 microns), percent by weight, *Max*	0·01

3.3 The material shall also comply with the requirements given in Table I. Tests shall be conducted according to the methods prescribed in Appendix B of this standard and to the methods prescribed in IS: 1683-1960.

References to the relevant clauses of Appendix B and IS: 1683-1960 are given in col 4 and 5 of the table respectively.

3.4 The colour imparted by the material in the rubber after vulcanization shall conform to that of the approved sample.

4. PACKING AND MARKING

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

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*See IS: 460-1953.

TABLE I REQUIREMENTS FOR NATURAL RED OXIDES OF IRON FOR RUBBER INDUSTRY

(*Clause* 3.3)

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST REF TO	
			Cl No. of Appendix B	Cl No. of IS: 1683-1960
(1)	(2)	(3)	(4)	(5)
i)	Specific gravity at 27°/27°C	5·0 to 5·5	—	C-3
ii)	Moisture content, percent by weight, *Max*	0·5	—	C-4
iii)	Loss on ignition, percent by weight, *Max*	1·0	B-2	—
iv)	Acidity in terms of sulphuric acid, percent by weight, *Max*	0·02	B-3	—
v)	Water soluble matter, percent by weight, *Max*	2	B-4	—
vi)	Siliceous matter, percent by weight, *Max*	5·0	B-5	—
vii)	*Iron oxide as ferric oxide* (Fe_2O_3), percent by weight, *Min*	90·0	B-6	—
viii)	Manganese (Mn) percent by weight, *Max*	0·05	—	C-7
ix)	Copper	To satisfy the requirements of the test	—	C-8

4.2 The packages shall be securely closed and marked with the name of the material; name of the manufacturer; recognized trade-mark, if any; weight of the material and the year of manufacture.

4.2.1 The packages may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act, 1952 and the Rules and Regulations made thereunder. Details of conditions, under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

APPENDIX A

(*Clause* 2.1)

SAMPLING OF NATURAL RED OXIDES OF IRON FOR RUBBER INDUSTRY

A-1. GENERAL REQUIREMENTS OF SAMPLING

A-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

A-1.1 Samples shall be taken in a protected place not exposed to damp air, dust or soot.

A-1.2 The sampling instrument shall be clean and dry when used.

A-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples, from adventitious contamination.

A-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

A-1.5 The samples shall be placed in clean, dry and air-tight glass or other suitable containers on which the material has no action.

A-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

A-1.7 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling and the year of manufacture of the material.

A-1.8 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

A-2. SCALE OF SAMPLING

A-2.1 Lot — All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute the lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots.

A-2.2 The number of containers to be selected from the lot shall depend upon the size of the lot and shall be in accordance with Table II.

TABLE II NUMBER OF CONTAINERS TO BE SELECTED

(*Clause* A-2.2)

LOT SIZE N (1)	NUMBER OF CONTAINERS TO BE SELECTED n (2)
Up to 100	5
101 to 300	6
301 „ 500	7
501 „ 800	8
801 „ 1 300	9
Above 1 300	10

A-2.3 These containers shall be selected at random from the lot and in order to ensure randomness, the procedure given in **A-2.3.1** may be adopted.

A-2.3.1 Arrange all the containers in the lot in a systematic manner and starting from any container count them as 1, 2, 3, ... up to r, r being the integral part of $\frac{N}{n}$

where

N = lot size, and

n = number of containers to be selected.

Every rth container, thus counted shall be withdrawn. All such containers shall constitute the sample.

A-3. TEST SAMPLES AND REFEREE SAMPLES

A-3.1 Draw with an appropriate sampling instrument small portions of the material from different parts of the container selected from the lot (*see* Table II). The total quantity of the material so drawn should be sufficient to conduct the tests for all the requirements but shall not exceed 500 g.

A-3.2 Mix thoroughly the portions drawn from the same container to form an individual sample representative of the container. In addition, mix together equal portions from the individual samples to form a composite sample representative of the lot.

A-3.3 Divide all the individual samples (*see* **A-3.1**) and the composite sample (*see* **A-3.2**) into three equal parts forming three sets of test samples.

Transfer these test samples immediately to thoroughly dried sample containers which shall then be sealed air-tight. Label them with all the particulars of sampling given under **A-1.7**. Send one of these sets to the purchaser and one to the supplier.

A-3.4 Referee Samples — The third set of test samples, bearing the seals of the purchaser and the supplier, shall constitute the referee samples to be used in case of dispute between the purchaser and the supplier. It shall be kept at a place as agreed to between the purchaser and the supplier.

A-4. NUMBER OF TESTS

A-4.1 Tests for fineness (*see* **3.2**), acidity, manganese and copper (*see* Table I) shall be conducted on each of the individual samples in the set of test samples.

A-4.2 Tests for specific gravity, moisture content, loss on ignition, water soluble matter, siliceous matter, and iron oxide shall be conducted on the composite sample in the set of test samples.

A-5. CRITERION FOR CONFORMITY

A-5.1 A lot shall be declared as conforming to this specification if the requirements of **A-5.2** to **A-5.4** are satisfied.

A-5.2 The individual samples when tested for copper shall satisfy the requirement for that characteristic as given in Table I.

A-5.3 The test results for fineness, acidity and manganese shall be recorded as shown in Table III. The mean and the range shall be calculated as follows:

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

Range (R) = The difference between the maximum and the minimum values of test result

The mean and the range shall be recorded as shown in col 4 and 5 of Table III respectively.

A-5.3.1 The corrected mean as given in col 6 of Table III shall be calculated. If the corrected mean satisfies the condition given in col 7 of Table III, the lot shall be considered to have satisfied the requirement of that characteristic.

A-5.4 The tests for specific gravity, moisture content, loss on ignition, water soluble matter, siliceous matter, and iron oxide shall satisfy the corresponding requirements for these characteristics as given in Table I.

TABLE III CRITERION FOR CONFORMITY

(*Clauses* A-5.3 *and* A-5.3.1)

SL No. (1)	CHARACTERISTIC (2)	TEST RESULTS (3)	MEAN (4)	RANGE (5)	CORRECTED MEAN (6)	CRITERION FOR CONFORMITY (7)
i)	Fineness :					
	a) Retained on *IS Sieve 8	1, 2, . . n	$\bar{X}_1$	R_1	$\bar{X}_1+0{\cdot}6\ R_1$	Corrected Mean $\leq 0{\cdot}4$
	b) Retained on *IS Sieve 15	1, 2, . . n	$\bar{X}_2$	R_2	$\bar{X}_2+0{\cdot}6\ R_2$	Corrected Mean $\leq 0{\cdot}01$
ii)	Acidity	1, 2, . . n	$\bar{X}_3$	R_3	$\bar{X}_3+0{\cdot}6\ R_3$	Corrected Mean $\leq 0{\cdot}02$
iii)	Manganese	1, 2, . . n	$\bar{X}_4$	R_4	$\bar{X}_4+0{\cdot}6\ R_4$	Corrected Mean $\leq 0{\cdot}05$

**See* IS : 460-1953

APPENDIX B

(*Clause* 3.3 *and Table* I)

CHEMICAL ANALYSIS OF NATURAL RED OXIDES OF IRON

B-1. QUALITY OF REAGENTS

B-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960) shall be employed in tests.

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

B-2. DETERMINATION OF LOSS ON IGNITION

B-2.1 Prepared Sample — Weigh about 10 g of the material in a previously weighed flat-bottomed dish about 6 cm in diameter. Keep the dish with the material in an oven. Maintain at 100° ± 2°C for two hours. At the end of this period, transfer the dish to a desiccator and cool. Remove the dish and weigh. Repeat the heating and weighing till the weight is constant.

B-2.2 Procedure — Weigh accurately about 3 g of the prepared sample (*see* **B-2.1**) in a previously ignited porcelain crucible of 50-ml capacity, and heat in a muffle furnace at 850°C. Cool to about 100°C and then allow the crucible and contents to cool in a desiccator. When room temperature is attained, weigh. Repeat the process until constant weight is obtained.

B-2.3 Calculation — Calculate the loss in weight as the percentage of the weight of dried sample taken for the test.

B-3. DETERMINATION OF ACIDITY

B-3.1 Reagents

B-3.1.1 *Standard Sodium Hydroxide Solution* — 0·02 N.

B-3.1.2 *Phenolphthalein Indicator* — Dissolve 0·5 g of phenolphthalein in 100 ml of rectified spirit (conforming to IS: 323-1959), which has been previously neutralized to the indicator.

B-3.2 Procedure — Weigh accurately about 20 g of the material into a 400-ml beaker and boil with 200 ml ammonia-free water for 5 min. Cool and make up to 250 ml in a 250-ml one-mark graduated flask (conforming to IS: 915-1958). Filter through a double filter paper (Whatman No. 40 or its equivalent) and reject the first 100 ml of the filtrate, collect the next 100 ml and titrate with standard sodium hydroxide solution using phenolphthalein as indicator.

B-3.3 Calculation

$$\text{Acidity (as } H_2SO_4\text{), percent by weight} = \frac{12{\cdot}25\, VN}{W}$$

where

V = the volume in ml of standard sodium hydroxide solution,

N = normality of standard sodium hydroxide solution, and

W = weight in g of the material taken for the test.

B-4. DETERMINATION OF WATER SOLUBLE MATTER

B-4.1 Procedure — Weigh accurately about 3 g of the prepared sample (*see* **B-2.1**). Wet it thoroughly with 3 ml of rectified spirit (conforming to IS: 323-1959) and add 200 ml of freshly boiled and cooled distilled water. Boil for 5 min and cool to room temperature. Make up the volume to 250 ml with distilled water. Shake and filter. Reject about 50 ml of the first portion of the filtrate and pipette out a 50-ml portion from the rest of the filtrate into a previously weighed porcelain dish. Evaporate to dryness on a water-bath. Dry the residue so obtained to constant weight in an oven at 100° ± 2°C.

B-4.2 Calculation

$$\text{Water soluble matter, percent by weight} = \frac{500\,w}{W}$$

where

w = weight in g of residue, and

W = weight in g of material taken for the test.

B-5. DETERMINATION OF SILICEOUS MATTER

B-5.1 Reagents

B-5.1.1 *Concentrated Hydrochloric Acid* — conforming to IS: 265-1950.

B-5.1.2 *Dilute Hydrochloric Acid* — 2 N and 0·1 N.

B-5.2 Procedure — Weigh accurately about 2 g of the material in a beaker. Add concentrated hydrochloric acid in a slight excess and digest in the fume cupboard. Add more acid if there is any effervescence on heating and continue digestion until all iron has been converted to a soluble chloride leaving greyish residue in suspension. Transfer to a porcelain basin and evaporate on a water-bath to dryness. After cooling, add about 100 ml of dilute hydrochloric acid (2 N) and digest on a water-bath for about 5 min. Filter the solution through a filter paper. Wash the residue 4 to 5 times with hot dilute hydrochloric acid (0·1 N) and finally with hot water till the wash liquid is free from chloride. Dry the residue in an oven, ash the filter paper and ignite the residue to constant weight.

B-5.3 Calculation — Calculate the weight of the residue as the percentage of the material taken for the test.

B-6. DETERMINATION OF FERRIC OXIDE

B-6.1 Method — Estimate ferric oxide volumetrically using a 0·1 N standard solution of potassium dichromate.

B-6.2 Reagents

B-6.2.1 *Concentrated Sulphuric Acid* — conforming to IS: 266-1950.

B-6.2.2 *Silica Gel*

B-6.2.3 *Concentrated Hydrochloric Acid* — conforming to IS: 265-1950.

B-6.2.4 *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 (sp-gr 1·16) and dilute with water to 200 ml.

B-6.2.5 *Mercuric Chloride Solution* — Dissolve 27 g of mercuric chloride in one litre of water.

B-6.2.6 *Phosphoric Acid* — sp-gr 1·7.

B-6.2.7 *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.

B-6.2.8 *Diphenylamine or Diphenyl Benzidine Indicator* — Dissolve one gram of diphenylamine or diphenyl benzidine in 100 ml of concentrated sulphuric acid. Use 3 drops (about 0·1 ml) for each titration.

B-6.2.9 *Standard Potassium Dichromate Solution* — 0·1 N. Finely powder about 6 g of potassium dichromate (analytical reagent quality) in a glass or agate mortar, and heat for 30 to 60 min in an air-oven at 140° to 150°C. Cool in a desiccator. Dissolve about 4·9 g of the powder, accurately weighed, in distilled water in a one-litre measuring flask and shake thoroughly. Make up to the litre mark.

$$\left(\frac{W}{49{\cdot}03}\right) N = \text{Strength of potassium dichromate solution}$$

where

W = weight of potassium dichromate dissolved in 1 000 ml of solution.

B-6.3 Procedure

B-6.3.1 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 100° ± 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 weight is obtained.

B-6.3.2 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 a hot-plate, evaporate 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 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 of diphenylamine or diphenyl benzidine indicator, and then titrate with standard potassium dichromate solution.

B-6.4 Calculation

$$\text{Ferric oxide, percent by weight} = \frac{7{\cdot}98\ VN}{W}$$

where

V = volume in ml of standard potassium dichromate solution,
N = normality of standard potassium dichromate solution, and
W = weight in g of the material taken for the test.