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Draft Indian Standard

WHITING FOR RUBBER INDUSTRY— SPECIFICATION

(Second Revision of IS 1685)

ICS 71.060.50

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 first revised in 1975. In the first revision, the requirements for calcium carbonate and matter soluble in water were added and requirements for pH and manganese content were modified. Further, the test methods common with other rubber compounding ingredients were deleted and reference were made to a separate standard for methods of sampling and test for rubber compounding ingredients.

Second 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 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 whiting for rubber industry.

2 REFERENCES

The following Indian Standards contains provision 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 the standard are encouraged to investigate the possibility of applying the most recent editions of the standard indicated below:

<i>IS No.</i>	<i>Title</i>
IS 264 : 2005	Nitric acid — Specification (<i>third revision</i>)
IS 2316 : 1990	Methods of preparation of standard solutions for colorimetric and volumetric analysis (<i>second revision</i>)
IS 1070 : 2023	Reagent grade water — Specification (<i>fourth revision</i>)
IS 7086 (Part 1) : 1973	Methods of sampling and test for rubber compounding ingredients, Part 1

3 REQUIREMENTS

3.1 Description

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

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

Table 1 Requirements of Whiting for Rubber Industry
(Clauses 3.2, 6.2.1 and 6.3.1.1)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Annex	Cl. No.in IS 7086 (Part 1)
(1)	(2)	(3)	(4)	(5)
i)	Sieve residue, percent by mass, <i>Max</i> : a) through 75-micron IS Sieve b) through 150-micron IS Sieve	0.4 0.01	— —	3
ii)	Relative density at 27 /27 °C	2.7 to 2.9	—	4
iii)	pH	8 to 11, or as agreed to between the purchaser and the supplier	—	5

iv)	Moisture content, percent by mass, <i>Max</i>	0.5	—	7
v)	Matter soluble in water, percent by mass, <i>Max</i>	0.5	—	8
vi)	Matter insoluble in hydrochloric acid, percent by mass, <i>Max</i>	3	—	9
vii)	Loss on ignition, other than moisture, percent by mass, <i>Max</i>	42 to 45	—	10
viii)	Manganese (as Mn), percent by mass, <i>Max</i>	0.03	—	11
ix)	Copper	To satisfy requirement of the test	—	12
x)	Alumina and iron oxide, percent by mass, <i>Max</i>	1	A	—
xi)	Calcium carbonate, percent by mass, <i>Min</i>	94	B	—

3.3 Compounding Test with Rubber

The base compound prepared with the material shall generally match in physical properties with those of the compound prepared in the same manner with the approved sample.

4 PACKING

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

5 MARKING

5.1 The packages shall be marked with the following:

- a) Name of the material;
- b) Manufacturer's name and trade-mark, if any;
- c) Net mass of the contents; and
- d) Year of manufacture.

5.2 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 Tests

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.

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 requirement of the specification if:

$$\bar{X} + 0.6 R \leq 0.03$$

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 the corresponding requirements.

ANNEX A [Table 1, Sl No. (x)]

DETERMINATION OF ALUMINA AND IRON OXIDE

A-1 Reagents

A-1.1 Ammonium Chloride

A-1.2 Ammonium Nitrate Solution — Approximately 2 percent (m/v)

A-1.3 Ammonium Hydroxide Solution — Relative density 0.90

A-1.4 Dilute Hydrochloric Acid — Approximately 25 percent (m/v)

A-1.5 Concentrated Nitric Acid — Relative density 1.42; conforming to IS 264

A-2 PROCEDURE

Weigh accurately about 2 g of the material dried to constant mass at (105 ± 2) °C and transfer to a 250 ml beaker. Add 50 ml of dilute hydrochloric acid, boil for 10 min and dilute to 100 ml. Filter through a filter paper (Whatman No. 40 or its equivalent). Wash the filter paper with hot water until the filtrate is free from chlorides. Collect the filtrate and washings in a beaker, add 2 g of ammonium chloride and then a few drops of concentrated nitric acid. Boil for 5 to 10 min. Render the contents slightly alkaline with ammonium hydroxide solution and bring to boil. Allow the beaker to stand in a warm place for half an hour and filter through a filter paper (Whatman No. 40 or its equivalent). Replace the beaker containing the filtrate by original beaker and dissolve the precipitate with a few millilitres of dilute hydrochloric acid followed by hot water. Add 2 g of ammonium chloride and precipitate with ammonium hydroxide solution. Boil and filter through a filter paper (Whatman No. 40 or its equivalent). Wash the precipitate with ammonium nitrate solution, dry with filter paper and ignite at about 850 °C.

A-3 CALCULATION

Express the mass of the residue as percentage of the mass of the dried material taken for the test.

ANNEX B

[Table 1, Sl No. (xi)]

DETERMINATION OF CALCIUM CARBONATE CONTENT

B-1 OUTLINE OF THE METHOD

The calcium carbonate content is determined volumetrically, using standard potassium permanganate solution.

B-2 REAGENTS

B-2.1 Standard Potassium Permanganate Solution — 0.1 N (*see 27* of IS 2316)

B-2.2 Bromine Water

B-2.3 Ammonium Oxalate Solution — Saturated

B-2.4 Dilute Hydrochloric Acid — 1 : 1 (v/v)

B-2.5 Dilute Sulphuric Acid — 1 : 4 (v/v)

B-3 PROCEDURE

Transfer to a beaker about 0.2 g of accurately weighed material previously dried to constant mass at (105 ± 2) °C. Dissolve the material in about 20 ml of dilute hydrochloric acid. Digest for 10 min on a steam-bath, dilute to 150 ml, filter and wash the residue with water. Add a few

millilitres of bromine water, heat to boiling and make the boiling solution ammoniacal. Filter off the residue, wash it thoroughly and reduce the filtrate by evaporation to 200 ml. To the slightly ammoniacal solution, heated to boiling, add an excess of hot ammonium oxalate solution. Continue boiling till the precipitate becomes granular. Allow to stand for 1 h, filter and wash with hot water. Pierce the apex of the filter paper with a stirring rod and wash the precipitate into a beaker with hot water. Pour warm dilute sulphuric acid through the paper and wash it a few times with warm acid. Add about 30 ml of dilute sulphuric acid, dilute to about 250 ml, heat to 60 °C and titrate with the standard potassium permanganate solution.

B-4 CALCULATION

$$\text{Calcium carbonate, percent by mass} = \frac{0.5004 V}{M}$$

where

V = volume in ml of potassium permanganate solution used for titration, and
 M = mass in g of the material taken for the test.