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भारतीय मानक मसौदा

रबर उद्योग के लिए हल्का मैग्नीशियम ऑक्साइड – विशिष्टि
(IS 9407 का पहला पुनरीक्षण)

Draft Indian Standard

LIGHT MAGNESIUM OXIDE FOR RUBBER INDUSTRY — SPECIFICATION

(First Revision of IS 9407)

ICS 71.060.20

Rubber and Rubber Products Sectional
Committee, PCD 13

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FOREWORD

(Formal clauses will be added later)

This standard was originally published in 1980.

For manufacturing products of good quality, it is essential that all the raw materials used in its production are of proper quality. This standard prescribes the quality of light magnesium oxide which is used in the rubber industry as an activator.

In this revision, the major changes are:

- a) Two types of light magnesium oxide have been included.
- b) Values of requirements of bulk density, moisture content, matter insoluble in hydrochloric acid, loss on ignition and magnesium oxide have been modified for the two types.
- c) Requirements for iodine adsorption have been removed and requirements for BET surface area have been incorporated.
- d) Amendment no.1 have also been incorporated. Additionally, the various editorial corrections, and references have been updated.

This standard contains clauses **4.3** and **5.1** which call for agreement between the purchaser and the supplier.

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 the methods of sampling and test for light magnesium oxide for rubber industry.

2 REFERENCES

The following Indian Standards contain 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/ISO No.</i>	<i>Title</i>
IS 265 : 2021	Hydrochloric acid — Specification (<i>fifth revision</i>)
IS 1675 : 1971	Specification for steric acid, technical (<i>first revision</i>)
IS 3399 : 2013	Zinc oxide for rubber industry — Specification (<i>third revision</i>)
IS 7086 (Part 1) : 1973	Methods of sampling and test for rubber compounding ingredients : Part 1
ISO 9277:2022	Determination of the specific surface area of solids by gas adsorption — BET method

3 TYPES

This standard prescribes following two types of light magnesium oxide:

- a) Type 1 — High purity light magnesium oxide; and
- b) Type 2 — Medium purity light magnesium oxide.

4 REQUIREMENTS

4.1 Description

The material shall be in the form of white, odourless, non-toxic free flowing powder.

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

Table 1 Requirement of Light Magnesium Oxide for Rubber Industry
(Clause 4.2)

Sl. No.	Characteristics	Requirement		Method of Test, for Ref to	
		Type 1	Type 2	Annex	Standard
(1)	(2)	(3)	(4)	(5)	(6)
i)	Bulk density, g/ml	0.35 to 0.45	0.50 to 0.60	A	—
ii)	Sieve residue, percent by mass, <i>Max</i>				Clause 3 of IS 7086 (Part 1)
	a) Through 53-micron IS Sieve	0.2	0.2		

	b) Through 150-micron IS Sieve	0.01	0.01		
iii)	Relative density 27 °C /27 °C	3.4 ± 0.2	3.4 ± 0.2	—	Clause 4 of IS 7086 (Part 1)
iv)	Moisture content, percent by mass, <i>Max</i>	0.5	1.0	—	Clause 7 of IS 7086 (Part 1)
v)	Matter soluble in water, percent by mass, <i>Max</i>	1.0	1.5	—	Clause 8 of IS 7086 (Part 1)
vi)	Matter insoluble in hydrochloric acid, percent by mass, <i>Max</i>	0.2	0.2	—	Clause 9 of IS 7086 (Part 1)
vii)	Loss on ignition, percent by mass, <i>Max</i>	8.0	8.0	—	Clause 10 of IS 7086 (Part 1)
viii)	Manganese (as Mn), percent by mass, <i>Max</i>	0.003	0.003	—	Clause 11 of IS 7086 (Part 1)
ix)	Copper (as Cu), percent by mass, <i>Max</i>	0.001	0.001	—	Clause 12 of IS 7086 (Part 1)
x)	Magnesium oxide, percent by mass, <i>Min</i>	98	96	B	—
xi)	Calcium oxide, percent by mass, <i>Max</i>	0.5	2	C	—
xii)	BET surface area, m ² /g, <i>Min</i>	140	140	—	ISO 9277
xiii)	Aluminium and iron oxide, percent by mass, <i>Max</i>	1.0	1.0	D	—

4.3 Compounding Test

If desired by the purchaser, the material may be compounded in polychloroprene rubber test recipe and the properties compared with the approved sample. The value for Mooney scorch obtained with the test sample shall not vary by more than 2 min from those obtained with the approved sample. The test recipe is given in Annex E.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed as agreed to between the purchaser and the supplier. Precautions shall be taken so as to protect it from moisture and moist air.

5.2 Marking

The following shall be marked on the package:

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

d) Month and year of manufacture.

5.2.1 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.

6 SAMPLING AND CRITERIA FOR CONFORMITY

6.1 Sampling

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

6.2 Number of Tests and Criteria for Conformity

Tests for all characteristics shall be conducted on the composite sample. The lot shall be considered as conforming to the specification if the composite sample satisfies each of these requirements.

ANNEX A

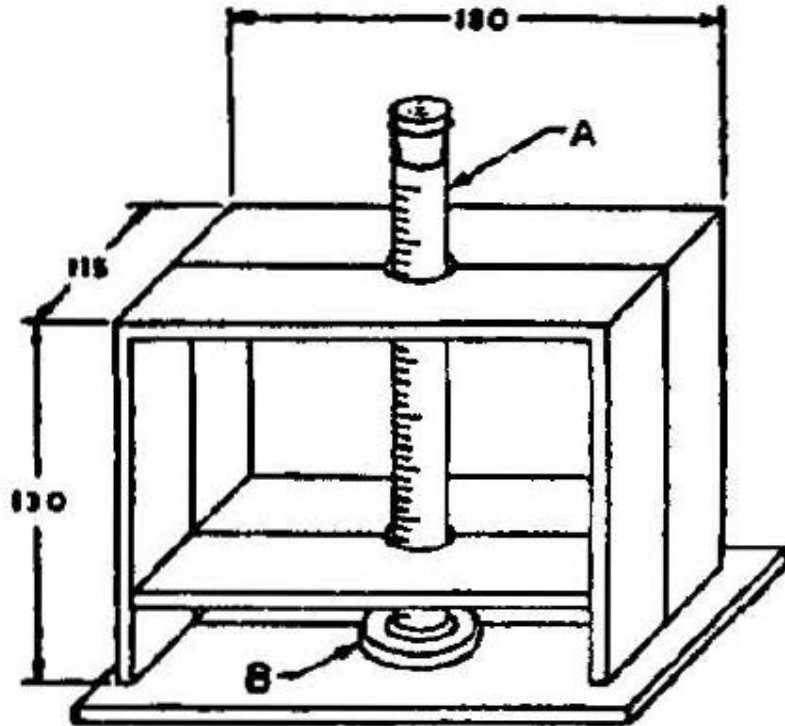
[Table 1, Sl. no.(i)]

DETERMINATION OF BULK DENSITY

A-1 APPARATUS

A-1.1 Assemble the apparatus as shown in Fig. 1. The base of the measurement cylinder *A* shall be ground flat and the empty measuring cylinder *A* together with the rubber bung shall weigh (250 ± 5) g. It shall be accurately calibrated to 250 ml with an error, if any, of less than 1 ml.

The distance between zero and 250 ml graduation on the measuring cylinder *A* shall be not less than 220 mm and not more than 240 mm. The distance between the flat-ground part of the base or measuring cylinder *A* and the rubber base pad *B*, when the measuring cylinder *A* is raised to the full height shall be (25 ± 2) mm.



All dimension in millimetres

FIG. 1 APPARATUS FOR THE DETERMINATION OF BULK DENSITY

A-1.2 Rubber Base Pad —The rubber base pad *B* shall have a hardness of 42 IRHD to 50 IRHD.

A-1.3 Balance — Pans of the balance shall be at least 10 cm in diameter and the balance shall be sensitive to less than 0.1 g.

A-2 PROCEDURE

A-2.1 Sieve about 40 g of the material through 250 micron IS Sieve on to a tared glazed paper and weigh it accurately. Slip the powder gently and smoothly into the measuring cylinder which should be held at 45° to the vertical, without knocking or squeezing. Assemble the apparatus as shown in Fig. 1. With the thumb and four fingers of one hand, gently grasp the upper part of the cylinder, and within one second lift it about 25 mm (taking care not to jerk the cylinder by knocking it against the upper stop) and let it drop. Continue lifting and dropping until 50 complete drops have been given to the cylinder. During this operation give a gentle turn of about 10° in the clockwise direction to the cylinder after every two drops. As soon as 50 drops are completed, raise the cylinder to eye level and read the volume of the material.

A-3 CALCULATION

A-3.1 Calculate bulk density at follows:

$$\text{Bulk density, g/ml} = \frac{m}{V}$$

where

m = mass in g of the material taken for the test, and

V = volume in ml of the material after 50 taps

ANNEX B
[Table 1, Sl.no. (x)]
DETERMINATION OF MAGNESIUM OXIDE

B-1 APPARATUS

B-1.1 Conical flask

B-1.2 Pipette

B-1.3 Burette

B-1.4 Watch glass

B-1.5 Balance

B-2 REAGENTS

B-2.1 Standard Sulphuric acid, 1 N

B-2.2 Standard Sodium Hydroxide Solution, 1 N, freshly standardized

B-2.3 Methyl Orange indicator Solution

Dissolve 0.01 g of methyl orange in 100 ml of water.

B-3 PROCEDURE

B-3.1 Weigh accurately about 1 g of the freshly ignited material and transfer it to a 250 ml conical flask. Add into the flask about 20 ml of water and transfer with a pipette 25 ml of standard sulphuric acid. Cover the flask with a watch glass and stir the flask carefully. Wash down the watch glass and the sides of the conical flask with water and titrate the solution in the flask with standard sodium hydroxide solution using methyl orange indicator. Carry out a blank determination using the same quantities of the reagents.

B-4 CALCULATION

$$\text{Magnesium oxide, percent by mass of the ignited residue} = \frac{2.016 (V_1 - V_2) N}{M} - 0.719A$$

where

V_1 = volume in ml of standard sodium hydroxide solution required for titration in the blank determination,

V_2 = volume in ml of standard sodium hydroxide solution required for titration in the test with the material,

N = normality of standard sodium hydroxide solution,

M = mass in g of the ignited material taken for the test, and

A = percentage by mass of calcium oxide as obtained in **C-4**

ANNEX C
[Table 1, Sl no. (xi)]
DETERMINATION OF CALCIUM OXIDE

C-1 APPARATUS

C-1.1 Beaker

C-1.2 Measuring cylinder

C-1.3 Balance

C-2 REAGENTS

C-2.1 Concentrated Hydrochloric Acid, *see* IS 265

C-2.2 Rectified Spirit, 95 percent (v/v).

C-2.3 Dilute Sulphuric Acid, 25 percent (m/v).

C-3 PROCEDURE

C-3.1 Dissolve about 0.5 g of freshly ignited material, accurately weighed, in concentrated hydrochloric acid added in small portions. Filter, if necessary. Add to the filtrate 100 ml of rectified spirit and 40 ml of dilute sulphuric acid and let stand overnight. If crystals of magnesium sulphate separate out, warm the mixture to about 50 °C to dissolve them. Filter, wash with a mixture of two volumes of rectified spirit and one volume of sulphuric acid and ignite at 650 °C. Weigh the residue.

C-4 CALCULATION

$$\text{Calcium oxide, percent by mass of the ignited residue} = \frac{41.18 A}{M}$$

where

A = mass in g of the residue, and

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

ANNEX D
[(Table 1, Sl No. (xiii)]
DETERMINATION OF ALUMINIUM OXIDE AND IRON OXIDE

D-1 APPARATUS

D-1.1 Beaker

D-1.2 Measuring cylinder

D-1.3 Crucible

D-1.4 Desiccator

D-1.5 Balance

D-2 REAGENTS

D-2.1 Ammonium Chloride

D-2.2 Ammonium Hydroxide Solution, 50 percent (v/v)

D-2.3 Ammonium Nitrate Solution, 2 percent (m/v)

D-2.4 Dilute Hydrochloric Acid, 50 percent (v/v)

D-2.5 Concentrated Nitric Acid

D-3 PROCEDURE

Weigh accurately about 2 g of the light magnesium oxide sample in a 400 ml beaker, add 50 ml of 50 percent hydrochloric acid, boil, dilute to 100 ml and filter. Wash the residue 5 times with 10 ml portions of hot distilled water. (The residue may be ignited and weighed for the estimation of insoluble siliceous matter). To the filtrate add 2 g of ammonium chloride, a few drops of concentrated nitric acid, boil for 10 min and cool. Add ammonium hydroxide until just alkaline, bring to boil, allow to stand for 10 min and filter the precipitate. Dissolve the residue into a new beaker by washing with 50 ml of hydrochloric acid followed by 5 hot distilled water washes of 10 ml each. More of hydrochloric acid and water may be used if the residue is undissolved. Add 2 g of ammonium chloride and re-precipitate with ammonium hydroxide, boil and filter. Wash with 5 portions of ammonium nitrate 10 ml each. Transfer residue to a pre-ignited tared crucible, ignite, cool in a desiccator and weigh.

D-4 CALCULATION

$$\text{Aluminium oxide and iron oxide, percent by mass} = \frac{m}{M} \times 100$$

where

m = mass in g of the residue, and

M = mass in g of the sample

ANNEX E

(Clause 4.3)

RECIPE FOR COMPOUNDING TEST

E-1 Test Recipe

The following test recipe is recommended for checking performance test requirements:

<i>Ingredient</i>	<i>Parts of Mass</i>
Polychloroprene rubber	100

Stearic acid (<i>see</i> IS 1675)	1
Semi-reinforcing furnace (SRF) carbon black	30
Zinc oxide (<i>see</i> IS 3399)	5
Magnesium oxide	4