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Draft Indian Standard

DOLOMITE FOR PAINT — SPECIFICATION

(First Revision of IS 15366)

(ICS 73.080; 87.060.10)

Raw materials for Paints, Varnishes and Related
Product Sectional Committee, CHD 21

Last Date for Comments: 24 November 2024

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FOREWORD

(Formal Clause shall be added later)

Dolomite, a naturally occurring mineral composed of calcium magnesium carbonate ($\text{CaMg}(\text{CO}_3)_2$), is generally utilized in the paint industry as a filler and extender. Standards on Dolomite for ceramic industry, glass industry and steel industry had been formulated; however, a need was felt to formulate a separate standard on dolomite for specific use of paint industry.

This standard was originally published in 2003. This first revision has been undertaken to align the product standards with current market demands, enhancing its acceptance and relevance.

Notable changes include:

- a) The product has been categorized into grades and types based on acid-insoluble impurities and median average particle size.
- b) A new requirement for median average particle size has been incorporated.
- c) To address health and safety concerns, restrictions on lead have been tightened, and restrictions for toxic heavy metals have been introduced.
- d) A suitable precautionary note has been added in the marking clause to prevent unforeseen events.

- e) Additionally, various editorial corrections and updates to references have been made where required.

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.

Draft Indian Standard
DOLOMITE FOR PAINT
— SPECIFICATION
(*First Revision*)

1 SCOPE

This standard prescribes requirements and methods of sampling and test for dolomite for use as extender in paint industry.

2 REFERENCES

The following standards contain provisions 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 this 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 33 : 1992	Method of sampling and test for inorganic pigments and extenders for paint (<i>third revision</i>)
IS 1070 : 1992	Reagent grade water (<i>third revision</i>)
IS 1303 : 1983	Glossary of terms relating to paints (<i>second revision</i>)

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 1303 and IS 33 shall apply.

4 REQUIREMENTS

4.1 Dolomite is natural calcium magnesium carbonate $[\text{CaMg}(\text{CO}_3)_2]$. The material shall be free flowing powder or in such a condition that it can be reduced to powder form by crushing without grinding action under a palette knife. The material shall be free from visible impurities.

4.2 Types

Ground dolomite is classified in two grades depending on the acid insoluble impurity in it and each grade is further classified in two types based on their particle size as given in Table 1.

- Grade A:** Natural calcium magnesium carbonate (dolomite) where acid insoluble as determined in Annex A is not more than 3 percent.

- b) **Grade B:** Natural calcium magnesium carbonate (dolomite) where acid insoluble as determined in Annex A is not more than 10 percent

4.3 Lead Restriction

The material shall be tested for restriction from lead in accordance with IS 101 (Part 8/Sec 5). When thus tested the material shall not contain lead or compounds of lead or mixtures of both, calculated as metallic lead exceeding 90 ppm (*see Note*).

4.4 Toxic Heavy Metal Restriction

Product shall not be manufactured using mercury and mercury compounds, cadmium, chromium VI, arsenic, antimony, and their oxides. The material shall not contain more than 0.1 percent by weight in total of above toxic heavy metals in the form of natural impurities or impurities entailed by the production process which are contained in the raw material when tested by the relevant Absorption/Emission Spectroscopic methods.

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

5 PACKING AND MARKING

5.1 Packing

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

5.2 Marking

The packages shall be securely closed and marked with following particulars:

- a) Name and type of the material;
- b) Name of the manufacturer or his recognized trade-mark;
- c) Mass of the material;
- d) Batch No. or Lot No. in code or otherwise; and
- e) Month and year of manufacture
- f) Expiry date/shelf life/best before
- g) Lead content (Maximum);
- h) Toxic heavy metals content
- j) A cautionary note as below:
 - a. Keep out of reach of children; or
 - b. This product may be harmful if swallowed or inhale

Table 1 Requirements for Dolomite for Paint Industry

(Clause 4.5)

SI No.	Characteristic	Requirements				Method of Test, Ref to	
		Grade A		Grade B		Annex	Clause of IS 33
		Type 1	Type 2	Type 1	Type 2		
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
i)	Volatile matter, percent by mass, <i>Max</i>	0.5	0.5	0.5	0.5	—	8
ii)	Residue on sieve, (500 mesh/25 micron),	0.1	1.0	0.1	1.0	—	9
iii)	Oil Absorption	14-20	14-20	12-16	12-16	—	10
iv)	pH Value	9-10	9-10	9-10	9-10		21
v)	Loss on Ignition percent by mass, <i>Max</i>	44-48	44-48	40-45	40-45	A-1	—
vi)	Acid insoluble residue, percent by mass, <i>Max</i>	3	3	10	10	A-2	—
vii)	Calcium Carbonate, percent by mass	45-55	45-55	43-50	43-50	A-3	—

viii)	Magnesium Carbonate, percent by mass	38-44	38-44	34-40	34-40	A-3	—
ix)	Median average particle size, at D 50, microns	3-7	NA	3-7	NA	A-4	—

5.3 BIS Certification Marking

The packages may also be marked with the Standard Mark.

5.3.1 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 there under, and the products may be marked with the Standard Mark.

6 SAMPLING

6.1 Representative samples of the material shall be drawn as prescribed under **5** of IS 33.

6.2 Criteria for Conformity

A lot shall be declared conforming to the requirements of this standard if the test results of the composite sample satisfy the requirements given in **4**.

7 TESTS

7.1 Tests shall be conducted according to the methods prescribed in various clause of IS 33 and various annexes to this standard.

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)

METHOD OF TEST

A-1 DETERMINATION OF LOSS ON IGNITION

A-1.1 Procedure

Weigh accurately about 2 g of the test sample, previously dried at $(100 \pm 2)^{\circ}\text{C}$ to constant mass into a platinum crucible; heat the crucible along with its contents at the temperature of $(1\ 000 \pm 25)^{\circ}\text{C}$ in a muffle furnace and ignite for 2 h. Weigh, repeat heating/cooling and weighing, (remove

crucible, place into desiccator, cool to room temp then weighing) till a constant mass is obtained. Express the loss in mass as the percentage of material taken for the test.

NOTE — Care should be taken to avoid hydration of the quick lime, formed during the process.

A-2 DETERMINATION OF ACID INSOLUBLE RESIDUE

A-2.1 Apparatus/Reagents

A-2.1.1 *Hydrochloric Acid* — 1: 1 v/v.

A-2.1.2 *Whatman Filter Paper No. 41*

A-2.1.3 *250 ml Beaker*

A-2.2 Procedure

Take about 5g of accurately weighed test sample in a clean 250 ml beaker and add 10 ml distilled water. Add 50 ml of 1 : 1 HCl slowly to it and heat it on a hot plate for about 30 min. Cool it to room temperature and filter it through previously dried and weighed Whatman filter paper No. 41. Wash the filter paper with distilled water till free from chlorides (this can be tested with AgNO₃ solution, white precipitate indicates presence of chlorides). Dry the filter papers in an electric oven at about 100°C to 105°C and weigh.

A-2.3 Calculation

$$\text{Acid insoluble matter, percent by mass} = \frac{100 (Y - X)}{\text{Mass of sample}}$$

where

X = mass of the empty filter paper, in g,

Y = mass of the filter paper + Acid insoluble residue, in g, and

$(X - Y)$ = mass of the acid insoluble residue, in g.

A-3 DETERMINATION OF CALCIUM AS CALCIUM CARBONATE AND MAGNESIUM AS MAGNESIUM CARBONATE

Two methods have been described for the estimation of calcium as calcium carbonate and magnesium as magnesium carbonate. Alternate method A-3.2 is quick and convenient to perform, however, method A-3.1 shall be used in case of dispute.

A-3.1 Calcium carbonate by permanganate titration and Magnesium carbonate by gravimetry

A-3.1.1 Reagents

A-3.1.1.1 *Dilute hydrochloric acid* — approximately 5 N.

A-3.1.1.2 *Ammonium oxalate solution* — saturated.

A-3.1.1.3 *Dilute ammonium hydroxide* — approximately 5 N and 1 N.

A-3.1.1.4 *Dilute potassium permanganate solution* — approximately 0.01 N.

A-3.1.1.5 *Dilute sulphuric acid* — approximately 5 N.

A-3.1.1.6 *Standard potassium permanganate solution* — 0.1 N.

A-3.1.1.7 *Sodium ammonium phosphate* — saturated.

A-3.1.1.8 *Ammonia* (2 percent)

A-3.1.2 *Preparation of Sample Solution*

Weigh accurately about 1 g of sample, previously dried at $(100 \pm 2)^\circ\text{C}$, into a platinum crucible and ignite as in A-2.1. Transfer the contents of the crucible completely to a 250 ml beaker with the help of water from a wash bottle jet. Rinse the crucible with a few millilitres of dilute hydrochloric acid to detach particles adhering to the crucible. Continue addition of hydrochloric acid to the beaker till a total of 40 ml of acid has been added. Heat the solution and boil for about 10 min. Cool and transfer completely to a 200 ml graduated flask, add water to make up the volume and mix thoroughly.

A-3.1.3 *Precipitation of Calcium*

Pipette out 50 ml of the prepared sample solution into a beaker to boiling and add 15 ml of water. Heat almost to boiling and add 15 ml of ammonium oxalate solution. To the hot solution, while constantly stirring, add dilute ammonium hydroxide (5N), dropwise, until the mixture is strongly alkaline. Let it stand for 2 h. Decant the supernatant liquid through a filter paper (Whatman No. 40) and preserve the liquid for estimation of magnesium.

A-3.1.4 *Estimation of Calcium*

Transfer all the precipitate from beaker to the filter paper by addition of cold water. The precipitate on the filter paper, is treated first with dilute ammonium hydroxide (1 N) and next with cold water until the filtrate does not decolorize dilute potassium permanganate solution in presence of dilute sulphuric acid. Puncture the filter paper and transfer the precipitate to a beaker with a fine jet of hot water. Add about 30 ml of dilute sulphuric acid, heat at 60°C and titrate with standard potassium permanganate solution. Towards the end of the titration introduce the filter paper which was used for filtration, into the titration vessel and carry out the titration till the end point is reached. Carry out a blank titration with all reagents, following the same procedure for the test but without the material.

A-3.1.5 *Calculation*

Calcium carbonate as (CaCO_3) ,

$$\text{percent by mass} = \frac{2.002(V_1 - V_2)N}{M}$$

where

V_1 = volume, in ml, of standard potassium permanganate solution used for the test;

V_2 = volume, in ml, of standard potassium permanganate solution used for the blank test;

N = normality of standard potassium permanganate solution; and

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

A-3.1.6 *Estimation of Magnesium*

The filtrate after recantation is concentrated to 100 ml. Add 20 ml of saturated solution of ammonium hydrophosphate, followed by 20 ml of ammonium hydroxide. The solution is stirred vigorously, using a rubber tipped glass rod and keep overnight. Precipitation of magnesium salt is carried by scratching of the solution. Filter the precipitate through Whatman No. 40 filter paper and wash thoroughly with 2 percent ammonia solution. Dry and ignite the residue in a previously weighed platinum crucible at $(1\ 000 \pm 25)^\circ\text{C}$ in a muffle furnace. After complete ignition cool and weigh the residue.

A-3.1.7 *Calculation*

Magnesium carbonate (MgCO_3),

$$\text{percent by mass} = \frac{3.030\ 8(a - b) \times 100}{M}$$

where

$(a - b)$ = weight of residue in g,

M = mass, in g, of the material for the test,

a = mass of the crucible and residue, in g, and

b = mass of empty crucible.

A-3.2 Calcium and magnesium carbonate by complexometric titration

A-3.2.1 *Reagents*

A-3.2.1.1 *Disodium EDTA* ($\text{Na}_2\text{H}_2\text{EDTA} \cdot 2\text{H}_2\text{O}$)

A-3.2.1.2 *Concentrated hydrochloric acid*

A-3.2.1.3 *Concentrated nitric acid*

A-3.2.1.4 *Ammonia-ammonium chloride buffer solution*, of pH 10: Dissolve 17.5 g of ammonium chloride in 142 ml of liquor ammonia (25%) and dilute to 250 ml with distilled water.

A-3.2.1.5 *NaOH solution, 10% (w/v).*

A-3.2.1.6 *Eriochrome Black T indicator (EBT), 0.4% methanolic solution: Grind a mixture of 0.05 g EBT with 5.0 g of NaCl. Use very small quantity of this indicator mixture for each titration (color should not be very dark).*

A-3.2.1.7 *Patton – Reeder’s indicator*

A-3.2.2 *Preparation of Sample Solution*

Weigh out accurately ~1.0 g of the sample in a 250 ml beaker. Moisten it with ~10 ml distilled water, ~10 ml conc. HCl and 1-2 drops conc. HNO₃. Heat the beaker gently ~80 °C for ~15 minutes (till the dissolution complete). Cool to room temperature. Transfer the solution quantitatively into a 250 ml volumetric flask by washing with distilled water. Make up the volume to the mark and mix uniformly and allow the SiO₂ to settle down (it will take ~30 minutes.)

A-3.2.3 *Standardization of EDTA Solution*

Weigh out 0.1 – 0.15 g of Zinc oxide in a 250 ml conical flask. Add 2 – 3 ml conc. HCl and shake to dissolve all the particles. Dilute with 40 – 50 ml distilled water and whirl the mixture. Neutralize the solution with liquor ammonia till the smell of ammonia persists. Add ~15 ml buffer solution and a pinch of EBT indicator (or 3 – 4 drops of the indicator solution), then the color of the solution turns wine red. Titrate the solution with standard 0.05 M EDTA solution till the wine-red color turns pure blue. Titrate slowly near the end point.

$$[EDTA] \text{ in } M = \frac{W_1 \times 1000}{81.4 \times V}$$

Where,

W₁ = Weight of zinc oxide in g

V = Volume of EDTA in ml

A-3.2.4 *Estimation of Total Calcium and Magnesium Carbonate*

Pipette out an aliquot of 25 ml of the stock solution prepared in A3.2.2 in a 250 ml conical flask. Add 1-2 drops of methyl orange indicator. The solution turns red (color should be very faint). Neutralize with drops of liquor ammonia to a just yellow color and then add 5 ml of NH₄OH + NH₄Cl buffer solution (pH 10) and a pinch of EBT indicator (or 3 – 4 drops of the indicator solution), when the color of the solution turns wine red. Titrate the solution with standard ~ 0.05 M EDTA solution till the wine red color turns pure blue. Titrate slowly near the end point. This titer value (V₁) gives total of (Ca²⁺ + Mg²⁺)

A-3.2.5 *Estimation of Calcium as Calcium Carbonate*

Pipette out an aliquot of 25 ml of the stock solution prepared in A3.2.2 in a 250 ml conical flask. Add 1 – 2 drops of methyl orange indicator. The solution turns red. Neutralize with drops of ~10% NaOH solution to a just yellow color and then add further 10 ml of the ~10% NaOH solution, mix well. Now add a pinch of Patton – Reeder’s indicator and shake. The color of the solution turns pink. Titrate the solution with standard 0.05 M EDTA solution till the pink color turns pure blue. Record the burette titer value (V_2) to estimate Ca^{2+} . The difference, ($V_1 - V_2$) estimates the amount of Mg^{2+} .

A-3.2.6 Calculation:

$$Ca \text{ as } CaCO_3 (\%) = \frac{100.08 \times V_2 \times S}{W}$$
$$Mg \text{ as } MgCO_3 (\%) = \frac{84.31 \times (V_1 - V_2) \times S}{W}$$

Where,

V_1 = Volume of standard EDTA solution, in ml, consumed for total of calcium and magnesium carbonate

V_2 = Volume of standard EDTA solution, in ml, consumed for calcium as calcium carbonate

S = Strength of EDTA solution in (M)

W = Weight of the sample taken in g

NOTE - Preparation of 1000 ml of 0.05 M EDTA solution: Weigh out 18.6 g disodium EDTA ($Na_2H_2EDTA \cdot 2H_2O$, MW = 372.22) in 1 liter amber color bottle. Add 500 ml distilled water and shake to dissolve. Add another 500 ml distilled water and shake well to make a homogeneous solution.

A-4 DETERMINATION OF MEDIAN AVERAGE PARTICLE SIZE BY LASER DIFFRACTION

A-4.1 General

Dolomite powder is dispersed in water and dilute dispersion of dolomite is passed through a laser diffraction particle size measurement chamber. Instrument produces a particle size distribution graph and calculates median average particle size (D 50) and other sizes like D 10, D 90 and D 97.

A-4.2 Reagents

A-4.2.1 Sodium Hexametaphosphate (SHMP) Solution – 0.1 % in distilled water / DM water

A-4.3 Procedure

A-4.3.1 Switch on the instrument and allow it to stabilize for 30 minutes. Start the measurement software on the computer or any other device attached to instrument. Wash the sample dispersing unit with distilled water/de-ionized water 3 to 4 times. Laser intensity value should be between 70 to 90%. Apply ultrasonication if required for cleaning. The final washing should be with dispersant solution. In dry condition, the laser obscuration value should be 0%. Fill the sample dispersing unit with 0.1% SHMP solution and set conditions as mentioned below for sample measurement. Carry out blank measurement first.

A-4.3.2 Set up following parameters in the test method (This may vary from make to make). Parameters listed here are guiding parameters for the machine.

Refractive index of material:	1.5694
Refractive index of Dispersant:	1.33
Absorbance:	0.0
Analysis model:	General purpose (or as defined in the make)
Particle shape:	Irregular
Sensitivity:	Normal
Stirring speed/pump speed:	3000 rpm
Obscuration (%):	9-13
Result unit:	Volume
Sonication Time:	1 min. with 100% power

A-4.3.3 Take approximately about 5 grams of powder sample in clean beaker and make a paste by drop wise addition of 0.1% sodium hexametaphosphate solution under continuous stirring with a sampling spatula. Consistency or viscosity of the paste should be like a honey. Using the spatula, slowly add this paste to sample dispersing unit of the instrument till a desired obscuration value is obtained. Sonicate the dispersion for 1 minute. Select the appropriate file from the software or set all essential parameters manually as per the manufacturer's standard operating procedure requirements as given in **A-4.3.2**. Report the median diameter $d(0.5)$ values as the median particle size of powder sample. The software should be set to report an average value of 3 readings. Other parameters as specified should be taken from particle size distribution.

NOTE

- 1) Follow operating instructions as mentioned by manufacturer.
- 2) After analysis, clean the dispersing unit several times with water as recommended by manufacturer.