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Indian Standard
**SPECIFICATION FOR
CALCIUM CARBONATE FOR EXPLOSIVE
AND PYROTECHNIC INDUSTRY**

(*First Revision*)

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TO

IS:7633-1982 SPECIFICATION FOR CALCIUM CARBONATE FOR EXPLOSIVE AND PYROTECHNIC INDUSTRY

(*First Revision*)

(Page 6, clause 5.3.1) - Substitute the following for the existing clause:

5.3.1 *Individual Samples* - For declaring the conformity of the lot to the requirements tested on individual samples (see 5.2.1), 5.3.1.1 and 5.3.1.2 shall be satisfied.

5.3.1.1 *Grit content* - The expression ' $\bar{x} + 0.6 R$ ' shall be less than or equal to the maximum specified limit given in Table 1 where \bar{x} = mean value of the test result and R = difference in maximum and minimum values of the test results.

5.3.1.2 *Fineness* - All the test results on each of the individual samples, shall meet the specified requirement given in Table 1.'

(CDC 51)

Indian Standard
SPECIFICATION FOR
CALCIUM CARBONATE FOR EXPLOSIVE
AND PYROTECHNIC INDUSTRY
(First Revision)

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Indian Standard
SPECIFICATION FOR
CALCIUM CARBONATE FOR EXPLOSIVE
AND PYROTECHNIC INDUSTRY
(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 31 May 1982 after the draft finalized by the Explosives and Pyrotechnics Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard was first published in 1975. The original standard covered only natural calcium carbonate in the form of calcite or limestone. In this revision, two types of the material have been specified, namely, natural and precipitated.

0.3 Calcium carbonate is used widely in propellant, pyrotechnic and other explosive compositions. This standard has been drawn up with a view to aiding the manufacturers of explosives and pyrotechnics in procuring material of requisite quality.

0.4 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*. 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 the methods of sampling and test for calcium carbonate used in explosive and pyrotechnic industry.

*Rules for rounding off numerical values (*revised*).

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2. TYPES

2.1 The material shall be of the following two types;

Type I Calcium carbonate, natural; and

Type II Calcium carbonate, precipitated.

3. REQUIREMENTS

3.1 Description — Type 1 of the material shall be in the form of powdered calcite or limestone. It shall be white to off-white in colour. Type II of the material shall be in the form of white powder.

3.1.1 The material shall be free from extraneous matter and grit.

3.2 The material shall comply with the requirements laid down in Table 1 when tested according to the methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in col 5 of the table.

TABLE 1 REQUIREMENTS FOR CALCIUM CARBONATE FOR EXPLOSIVE AND PYROTECHNIC INDUSTRY

SL No.	CHARACTERISTIC	REQUIREMENT FOR		METHOD OF TEST (Ref to Cl No. in Appendix A)
		Type I	Type II	
(1)	(2)	(3)	(4)	(5)
i)	Loss on drying, percent by mass, <i>Max</i>	0.5	0.5	A-2
ii)	Calcium carbonate (as CaCO ₂) (on dry basis), percent by mass, <i>Min</i>	98.0	98.0	A-3
iii)	Matter insoluble in dilute hydrochloric acid, percent by mass, <i>Max</i>	0.6	0.2	A-4
iv)	Fineness (material retained on 125-micron* IS Sieve) percent by mass	Nil	Nil	A-5
v)	Water soluble alkali (as Na ₂ O) percent by mass, <i>Max</i>	0.03	0.05	A-6
vi)	Grit, percent by mass, <i>Max</i>	0.06	0.02	A-7
vii)	Chlorides (as Cl), percent by mass, <i>Max</i>	0.05	0.04	A-8
viii)	Total water soluble matter, percent by mass, <i>Max</i>	0.5	0.5	A-9

*90-micron for material required for use in propellents.

3.3 Additional Requirements for Material Required for use in Propellents — The material when required for use in propellents, shall also comply with the requirements laid down in Table 2 in addition to the requirements specified in Table 1.

TABLE 2 ADDITIONAL REQUIREMENTS FOR CALCIUM CARBONATE FOR USE IN PROPELLENTS

SL No.	CHARACTERISTIC	REQUIREMENT FOR		METHOD OF TEST (Ref to Cl No. in Appendix A)
		Type I	Type II	
(1)	(2)	(3)	(4)	(5)
i)	Magnesium compounds (as $MgCO_2$), percent by mass, <i>Max</i>	0.5	0.5	A-10
ii)	Iron (as Fe), percent by mass, <i>Max</i>	0.05	0.05	A-11
iii)	Settling test (volume occupied by 5 g), ml	4 to 10	4 to 6	A-12

4. PACKING AND MARKING

4.1 Packing — Unless otherwise agreed to between the purchaser and the supplier, the material shall be packed in clean jute bags with a polyethylene liner.

4.2 Marking — The packages shall be marked legibly and indelibly with the following information:

- a) Name and type of the material;
- b) Net mass of the material;
- c) Manufacturer's name and/or his recognized trade-mark, if any;
- d) Year of manufacture; and
- e) Lot number in code or otherwise to enable the batch of manufacture to be traced back from records.

4.3 BIS Certification Marking

The product may also be marked with Standard Mark.

4.3.1 The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5. SAMPLING

5.1 Preparation of Test Samples — Representative samples of the material shall be prepared as prescribed in Appendix B of IS : 918-1968*.

5.2 Number of Tests

5.2.1 Tests for the determination of grit content and fineness shall be conducted on each of the individual samples.

5.2.2 Tests for the determination of all the remaining characteristics given in Tables 1 and 2 shall be carried out on the composite sample.

5.3 Criteria for Conformity

5.3.1 For Individual Samples — For declaring the conformity of the lot:

$\bar{x} + 0.6 R$ shall be less than or equal to the maximum specified requirements

where

\bar{x} = mean value of the test results, and

R = range of results.

5.3.2 For Composite Samples — For declaring the conformity of the lot to the requirements of all the characteristics tested on the composite sample, the test results shall satisfy the corresponding specified requirements.

APPENDIX A

(Clause 3.2 and Table 2)

METHODS OF TEST FOR CALCIUM CARBONATE FOR EXPLOSIVE AND PYROTECHNIC INDUSTRY

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (see IS : 1070-1977†) shall be employed in tests.

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

*Specification for calcium carbonate, precipitated, for cosmetic industry (first revision).

†Specification for water for general laboratory use (second revision).

A-2. DETERMINATION OF LOSS ON DRYING

A-2.1 Procedure — Weigh accurately about 5 g of the material in a weighed, clean and dry squat form weighing bottle, and dry to constant mass at 100° to 105°C.

A-2.2 Calculation

$$\text{Loss on drying, percent by mass} = 100 \times \frac{M_1}{M_2}$$

where

M_1 = loss in mass in g on drying, and

M_2 = mass in g of the material taken for the test.

A-3. DETERMINATION OF CALCIUM CARBONATE

A-3.0 Methods — Two methods have been prescribed for this test, namely, Method A and Method B. Method A is classical while Method B is complexometric method. In case of dispute Method A shall be followed.

A-3.1 Method A

A-3.1.0 Outline of the Method — Calcium is precipitated as its oxalate; the precipitate is dissolved in dilute sulphuric acid and the solution is titrated against permanganate solution.

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-3.1.2 Procedure — Weigh accurately about 1 g of the material, dried as under **A-2.1** and transfer it to a 250-ml beaker. Moisten thoroughly with a few millilitres of water and add dilute hydrochloric acid dropwise until solution is complete. Continue adding the acid until a total of 40 ml has been added. Heat the solution and boil for about 10 minutes. Cool and transfer completely to a 200-ml graduated flask, add water to make up the volume and mix thoroughly. Pipette out 50 ml of this solution to

a beaker and add 75 ml of water. Heat almost to boiling and add 15 ml of ammonium oxalate solution. To the hot solution, constantly stirred, add dilute ammonium hydroxide (5 N) dropwise until the mixture is strongly alkaline. Let it stand for two hours. Decant the clear supernatant liquid through a filter paper and then completely transfer the precipitate to the filter paper with cold water. Wash the precipitate first with dilute ammonium hydroxide (1 N) and then with cold water until the filtrate does not decolorize hot 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 test with all reagents following the procedure for the test but without the material.

A-3.1.3 Calculation

$$\text{Calcium carbonate (as CaCO}_3\text{), percent by mass} = \frac{20 \cdot 02 (V_1 - V_2) N}{M}$$

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 taken for the test.

A-3.2 Method B

A-3.2.0 Outline of the Method — The solution of the material, after adjustment of pH, is titrated with EDTA solution using Patton and Reader's indicator.

A-3.2.1 Reagents

A-3.2.1.1 Dilute hydrochloric acid — approximately 5 N.

A-3.2.1.2 Dilute sodium hydroxide solution — approximately 1 N.

A-3.2.1.3 Standard calcium carbonate solution — 0.01 N. Weigh 0.5004 g of calcium carbonate, dried as in A-2.1 and dissolve in the minimum quantity of dilute hydrochloric acid. Make up the volume with water to 1 litre.

A-3.2.1.4 Patton and Reader's indicator [2-hydroxy-1-(2-hydroxy-4-sulpho-1-naphthylazo)-3-naphthoic acid] — Triturate 0.5 g of the dyestuff with 50 g of anhydrous sodium sulphate in a glass mortar until a homogeneous mixture is obtained.

A-3.2.1.5 Standard disodium ethylenediamine tetra acetate (EDTA) Solution — Weigh 3.7 g of disodium ethylenediamine tetra acetate dihydrate and dissolve in water. Make up the volume to 1 litre. Standardize the solution against standard calcium carbonate solution as under **A-3.2.3**.

A-3.2.2 Prepared Sample Solution — Weigh accurately about 3 g of the material, dried as under **A-2.1** and transfer it to a beaker. Moisten thoroughly with a few millilitres of water and add dilute hydrochloric acid dropwise until the solution is complete. Continue adding the acid until a total of 50 ml has been added. Heat the solution and boil for about 10 minutes. Cool and transfer quantitatively to a 500-ml graduated flask. Pipette out 100 ml of this solution and dilute further to 1 litre. Use this diluted solution for the test under **A-3.2.3**.

A-3.2.3 Procedure — Pipette out 10 ml of the *prepared sample* solution (*see A-3.2.2*) into a 250-ml conical flask. Add 25 ml of water and 10 ml of dilute sodium hydroxide solution (pH of mixture should be greater than 12). Add 0.5 g of Patton and Reader's indicator and titrate with standard EDTA solution until the colour changes from wine red to distinct blue.

A-3.2.4 Calculation

Calcium carbonate (as CaCO₃),
percent by mast =
$$\frac{50.04 \times 50 \times NV}{M}$$

where

- V = volume in ml of standard EDTA solution,
- N = normality of standard EDTA solution, and
- M = mass in g of the material taken for the test in **A-3.2.2**.

A-4. DETERMINATION OF MATTER INSOLUBLE IN HYDROCHLORIC ACID

A-4.1 Reagents

A-4.1.1 Dilute Hydrochloric Acid – approximately 2 N.

A-4.2 Procedure — Weigh accurately about 2 g of the material. Transfer it to a round bottom flask with 10 ml of dilute hydrochloric acid. Fix up a reflux condenser and boil the material on a sand-bath for 5 minutes. Cool and filter through a sintered glass crucible No. G 4. Wash free

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from chlorides, dry the residue in an oven at 105 to 110°C to constant mass, cool and weigh.

A-4.3 Calculation

$$\begin{array}{l} \text{Matter insoluble in hydrochloric} \\ \text{acid, percent by mass} \end{array} = 100 \times \frac{M}{M_1}$$

where

M = mass in g of the residue, and

M_1 = mass in g of the material taken for the test.

A-5. DETERMINATION OF FINENESS

A-5.1 Procedure — Place 25.0 g of the material, accurately weighed, on to a 125-micron IS Sieve and wash the material on the sieve with water, gently brushing with a soft camel-hair brush, until the washings are clear. Dry the residue to constant mass at $100 \pm 2^\circ\text{C}$.

A-5.1.1 For the material required for use in propellents, 90-micron IS Sieve, shall be used.

A-5.2 Calculation

$$\begin{array}{l} \text{Fineness (material retained on 125-micron} \\ \text{IS Sieve), percent by mass} \end{array} = 4 M$$

where

M = mass in g of the residue on the sieve.

A-6. DETERMINATION OF WATER SOLUBLE ALKALI

A-6.0 Outline of the Method — A suspension of the material in water is titrated with hydrochloric acid using phenolphthalein indicator. The end point is verified after keeping the suspension for a total of 5 hours.

A-6.1 Reagents

A-6.1.1 Phenolphthalein Indicator Solution — Dissolve 0.1 g of phenolphthalein in 60 ml of rectified spirit (*see* IS : 323-1959*) and dilute with water to 100 ml.

A-6.1.2 Standard Hydrochloric Acid — 0.1 N.

A-6.2 Procedure — Weigh accurately about 10 g of the material in a 250-ml beaker, add about 100 ml of carbon dioxide-free water and 10 drops of phenolphthalein indicator. Stir and allow to stand for one hour. Filter and immediately titrate with standard hydrochloric acid till disappearance of pink colour. Allow to stand for one hour, stir again and if the pink colour develops again, titrate further with standard hydrochloric acid. Repeat 4 times, at intervals of 1 hour.

*Specification for rectified spirit (*revised*).

A-6.3 Calculation

$$\text{Soluble alkali (as Na}_2\text{O) , percent by mass} = \frac{3 \cdot 1 V N}{M}$$

where

V = total volume in ml of standard hydrochloric acid used for the titration(s),

N = normality of standard hydrochloric acid, and

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

A-7. DETERMINATION OF GRIT

A-7.0 Outline of the Method — The portion retained on 75-micron IS Sieve is boiled with aqua regia. The insoluble residue obtained after digestion with acid is dried. The dried residue is then sieved through 125-micron IS Sieve and the portion retained on the sieve is weighed. If the entire residue (retained on 125-micron IS Sieve) scratches soda glass then the mass, previously obtained, is expressed as percentage grit.

A-7.1 Apparatus

A-7.1.1 Sieves — 75-micron and 125-micron IS Sieve.

A-7.1.2 Glass Slides — soda glass.

A-7.2 Reagents

A-7.2.1 Dilute Hydrochloric Acid — approximately 5 N.

A-7.2.2 Aqua Regia — prepared by mixing 3 volumes of concentrated hydrochloric acid and one volume of concentrated nitric acid.

A-7.3 Procedure

A-7.3.1 Weigh accurately about 50 g of the material and sieve through 75-micron IS Sieve with a 25-mm brush. Take the portion retained on the sieve in a 250-ml beaker and add to it slowly and carefully about 100 ml of dilute hydrochloric acid. After the initial action has subsided, boil it for 20 minutes. Cool the beaker and decant the diluted acid carefully without losing any residue remaining at the bottom of the beaker.

A-7.3.2 Boil the residue obtained in **A-7.3.1** with about 50 ml of aqua regia (covering the beaker with a watch-glass) for about 30 minutes. Cool the beaker, dilute the acid with water, and then decant the diluted acid carefully without losing any residue remaining at the bottom of the beaker. Wash the residue three times with water and dry the residue in an oven.

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A-7.3.3 Sieve the dried residue carefully through 125-micron IS sieve with a light camel-hair brush. Weigh accurately the portion retained on the sieve and carry out the soda glass scratching test, as described in **A-7.5**. If the residue scratches soda glass express the mass (previously obtained) as percentage grit.

NOTE — Care should always be taken not to lose any material at any stage of the test.

A-7.4 Calculation

$$\text{Grit, percent by mass} = 100 \times \frac{M}{M_1}$$

where

M = mass in g of the residue scratching soda glass, and

M_1 = mass in g of the material taken for the test.

A-7.5 Soda Glass Scratch Test — Take a portion of the residue (retained on 125-micron IS Sieve) in between the two clean glass slides and press the slides by hand along the length and breadth of the glass. Examine whether any scratch in the glass is obtained. If there is any scratch in the glass then the residue is termed as grit.

A-8. DETERMINATION OF CHLORIDES

A-8.0 Outline of the Method — The turbidity produced with silver nitrate solution is matched against that obtained with standard chloride solution.

A-8.1 Apparatus

A-8.1.1 Nessler Cylinders — 50 ml capacity.

A-8.2 Reagents

A-8.2.1 Concentrated Nitric Acid — See IS : 264-1976*.

A-8.2.2 Standard Sodium Chloride Solution — Dissolve 1.649 g of sodium chloride in water and make up the volume to 1 000 ml. Pipette out 10 ml of the solution, dilute with water and make up the solution to 1 000 ml. One millilitre of this solution contains 0.01 mg of chloride (as Cl).

A-8.2.3 Silver Nitrate Solution — approximately 2 percent (m/v).

A-8.3 Procedure — Dissolve 1.0 g of the material in water by the addition of 3 ml of concentrated nitric acid and boil cautiously to remove carbon dioxide, cool and make up the volume to 100 ml. Pipette out 10 ml of this solution into a Nessler cylinder and add 1 ml of silver nitrate

*Specification for nitric acid (second revision).

solution. Carry out a control test in the other Nessler cylinder using 5 ml and 4 ml of standard sodium chloride solution respectively for Type I and Type II of the material and the same quantities of other reagents. Stir both the solutions with glass rods and compare the turbidity produced in the two cylinders after 5 minutes.

A-8.3.1 The limit prescribed shall be taken as not having been exceeded if the turbidity produced in the test with the material is not greater than that produced in the control test.

A-9. DETERMINATION OF WATER SOLUBLE MATTER

A-9.0 Procedure — Treat 15 g of the material with 300 ml of freshly boiled water. Boil gently in a covered vessel for 15 minutes. Cool to room temperature and filter. Wash the filter paper twice with 15 ml of water. Make up the volume to 500 ml in a standard flask. Take 100 ml of the filtrate and evaporate to dryness in a tared porcelain dish (M_1) on a water bath. Dry for one hour at 103 to 105°C. Cool in a desiccator and weigh (M_2). Calculate the water soluble matter as follows:

$$\text{Water soluble matter, percent by mass} = (M_2 - M_1) \times \frac{100}{M}$$

where

M_2 = mass in g of the dish and residue,

M_1 = mass in g of the empty dish, and

M = mass in g of the material present in the aliquot.

A-10. DETERMINATION OF MAGNESIUM COMPOUNDS

A-10.0 Outline of the Method — The solution obtained after precipitation of oxalates in ammoniacal medium is evaporated to dryness, the residue obtained is ignited and weighed.

A-10.1 Reagents

A-10.1.1 Concentrated Hydrochloric Acid — see IS : 265-1976*.

A-10.1.2 Oxalic Acid Solution — 6 percent.

A-10.1.3 Methyl Red Indicator Solution — Dissolve 0.03 g of methyl red in 100 ml of water.

A-10.1.4 Ammonium Hydroxide — 1 : 1 (v/v).

A-10.1.5 Concentrated Sulphuric Acid — see IS : 266-1977†.

*Specification for hydrochloric acid (second revision).

†Specification for sulphuric acid (second revision).

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A-10.2 Procedure — Weigh accurately 1 g of the material in a beaker, add to it 140 ml of water and 5 ml of concentrated hydrochloric acid. Heat and then boil for 1 minute. To the hot solution add rapidly 40 ml of oxalic acid solution and stir vigorously until precipitation is well established. Add immediately to the warm mixture 2 drops of methyl red indicator and then add dropwise ammonium hydroxide until the mixture is just alkaline. Cool to room temperature, transfer to a 100-ml graduated cylinder, dilute with water to 100 ml, mix and let stand for 4 hours. Filter, take the filtrate in a weighed platinum dish and add to it 0.5 ml of concentrated sulphuric acid. Evaporate the mixture on a steam-bath to a small volume. Then carefully heat over a small flame to dryness and then continue heating to complete decomposition and volatilization of ammonium salts. Cool in a desiccator and weigh. Repeat heating, cooling and weighing until constant mass (± 1 mg) is obtained.

A-10.3 The material shall be taken to have passed the test if the mass of the residue does not exceed 5 mg.

A-11. TEST FOR IRON

A-11.0 Outline of the Method — The colour produced with thioglycollic acid in ammoniacal medium is compared with that obtained with standard iron solution.

A-11.1 Apparatus

A-11.1.1 Nessler Cylinders — 50 ml capacity.

A-11.2 Reagents

A-11.2.1 Concentrated Hydrochloric Acid — see IS : 265-1976*.

A-11.2.2 Citric Acid Solution — approximately 20 percent.

A-11.2.3 Thioglycollic Acid

A-11.2.4 Dilute Ammonium Hydroxide — approximately 5 N.

A-11.2.5 Standard Iron Solution — Dissolve 0.702 g of ammonium ferrous sulphate [$\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 7\text{H}_2\text{O}$] in water containing 10 ml of dilute sulphuric acid. Transfer to a 1 000-ml graduated flask and dilute with water to the mark. Pipette out 10 ml of this solution into a 100-ml graduated flask and dilute this with water to the mark. One millilitre of this solution contains 0.01 mg of iron (as Fe).

A-11.3 Procedure — Dissolve 1.0 g of the material in a mixture of 25 ml of water and 2.5 ml of concentrated hydrochloric acid. Boil, dilute with

*Specification for hydrochloric acid (*second revision*).

water and make up the volume in a volumetric flask to 200 ml. Transfer by pipette 40 ml of this solution to a Nessler cylinder. Add 2 ml of citric acid solution and 2 drops of thioglycollic acid. Mix and make the solution alkaline with dilute ammonium hydroxide. Make up the volume to 50 ml mark. Carry out a control test in another Nessler cylinder using 10 ml of standard iron solution in place of the material and the same quantities of other reagents. Compare the colour produced, if any, after 5 minutes.

A-11.3.1 The limit prescribed shall be taken as not having been exceeded if the intensity of colour produced in the test with the material is not greater than that produced in the control test.

A-12. SETTLING TEST

A-12.1 Procedure — Introduce 5.0 g of the material into a graduated tube 55 cm long and 1.6 cm in internal diameter and add water up to 100-ml mark. Shake the mixture and allow it to settle in a vertical position on a bench which is free from vibration. After allowing to stand for 1 hour, take the readings of the sharp line between the chalk and the supernatant liquid.

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002
 Telephones: 323 0131, 323 8375, 323 9402
 Fax : 91 11 3234062, 91 11 3239399

Telegrams: Manaksanstha
 (Common to all Offices)

Central Laboratory:

Ptot No. 20/9, Site IV, Sahibabad Industrial Area, Sahibabad 201010 Telephone
8-77 00 32

Regional Offices:

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*Eastern : 1/14 CIT Scheme VII M, V.I.P. Road, Manikola, CALCUTTA 700054 337 86 62

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E-52, Chitaranjan Marg, C-Scheme, JAIPUR 302001 37 29 25

117/418 B, Sarvodaya Nagar, KANPUR 208005 21 68 76

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