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SPECIFICATION FOR ACTIVATED CALCIUM CARBONATE FOR RUBBER INDUSTRY

(*First Revision*)

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Indian Standard

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(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 1 September 1976, after the draft finalized by the Rubber Products Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard was first published in 1958. In this revision, requirement for insoluble siliceous matter has been removed while the maximum limit for moisture content has been lowered. New requirements for *p*H and hydrochloric acid insolubles have been added. Further, wetting and compounding tests have also been included in the standard. Instead of leaving the requirement for total fatty matter to be decided between the purchaser and the supplier, a range has been specified.

0.3 This standard contains clauses **2.4** and **3.1** which call for agreement between the purchaser and the supplier.

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 methods of sampling and test for activated calcium carbonate intended for use in rubber industry.

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

2. REQUIREMENTS

2.1 Description — The material shall consist of precipitated calcium carbonate coated with thin layer of fatty acids or their salts. It shall be in the form of a free-flowing, white powder, free from visible impurities.

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

TABLE 1 REQUIREMENTS FOR ACTIVATED CALCIUM CARBONATE

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Appendix	Cl No. in IS : 7086 (Part I)-1973*
(1)	(2)	(3)	(4)	(5)
i)	Sieve residue on 150-micron IS Sieve, percent by mass, *Max*	0·01	—	3
ii)	*p*H	10·5 ± 0·5	—	5
iii)	Hydrochloric acid insolubles, percent by mass, *Max*	0·2	—	9
iv)	Loss on ignition, percent by mass	43 to 46	—	10
v)	Manganese (as Mn), percent by mass, *Max*	0·02	—	11
vi)	Copper (as Cu), percent by mass, *Max*	0·005	—	12
vii)	Total fatty matter, percent by mass	2·0 to 3·5	A	—
viii)	Mixed oxides, percent by mass, *Max*	1·0	B	—
ix)	Chlorides (as Cl), percent by mass, *Max*	0·04	C	—
x)	Calcium carbonate and magnesium carbonate together (as $CaCO_3$), percent by mass, *Min*	92·0	D	—
xi)	Moisture content, percent by mass, *Max*	0·6	E	—

*Methods of sampling and test for rubber compounding ingredients, Part I.

2.3 Wetting Test — When about 1 g of the material is shaken with 10 ml of water in a test tube, the material shall float on the surface.

2.4 Compounding Test — If desired by the purchaser, the material may be compounded in natural rubber test recipe and the properties compared with the approved sample. The values obtained with the test sample shall not vary by more than ± 10 percent for tensile strength, elongation at break and hardness and not more than ± 15 percent for modulus at 300 percent elongation and tear strength from those obtained with the approved sample. The test recipe is given in Appendix F.

3. PACKING AND MARKING

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

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

3.3 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 and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. 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.

4. SAMPLING AND CRITERIA FOR CONFORMITY

4.1 Sampling — Sampling shall be done as prescribed in 15 of IS : 7086 (Part I)-1973*.

4.2 Number of Tests

4.2.1 Tests for manganese and copper shall be conducted on individual samples.

4.2.2 Tests for all other characteristics shall be conducted on composite sample.

4.3 Criteria for Conformity

4.3.1 *For Individual Samples* — The mean and range of the test results for manganese and copper 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.

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*Methods of sampling and test for rubber compounding ingredients, Part I.

The lot shall be deemed to have satisfied the requirements of the specification if $\overline{X} + 0{\cdot}6 \ R \leqslant 0{\cdot}02$ for manganese and $\leqslant 0{\cdot}005$ for copper.

4.3.2 *For Composite Sample* — In respect of all other characteristics, the lot shall be considered as conforming to the specification if the composite sample satisfies each of these requirements.

APPENDIX A

[*Table* 1, *Item* (vii)]

DETERMINATION OF TOTAL FATTY MATTER

A-1. REAGENTS

A-1.1 Ether

A-1.2 Dilute Hydrochloric Acid — approximately 5 N.

A-1.3 Acetone

A-2. PROCEDURE

A-2.1 Weigh accurately about 5 g of the material in a 400-ml beaker and wet the material by adding a little ether. Cover with a cover-glass and carefully add (a few millilitres at a time) 50 ml of dilute hydrochloric acid. Boil, dilute to about 100 ml with water and cool to room temperature. Transfer to a 300-ml separating funnel, rinse out the beaker with 25 ml of ether and transfer to the separating funnel. Shake gently, release the pressure by inverting the funnel and gradually opening the tap. Allow the two layers to separate completely, then run off the aqueous layer into the original beaker. Filter the ether layer through a dry filter paper into a tared conical flask. Transfer the aqueous layer back into the separating funnel and extract twice as before but using 15 ml portions of ether. *Reserve the aqueous layer for the tests prescribed in Appendix B.* Filter the combined ether extracts also through the same filter paper and collect the filtrate in the tared conical flask. Finally wash the filter paper with ether, distil the ether until only about 5 ml remain in the flask, then add about 5 ml of acetone and evaporate the mixture on a water-bath. Dry the residue at 60°C. In order to reduce the drying time to about 15 minutes and minimize volatilization and oxidation of fatty acid, blow through the flask a gentle current of air two or three times during drying. Cool the flask in a desiccator and reweigh.

A-3. CALCULATION

$$\text{Total fatty matter, percent by mass} = \frac{100\,(A - B)}{M}$$

where

A = mass in g of the conical flask containing the dried residue,

B = mass in g of the conical flask, and

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

APPENDIX B

[*Table* 1, *Item* (viii)]

DETERMINATION OF MIXED OXIDES

B-1. REAGENTS

B-1.1 Concentrated Nitric Acid — relative density 1·42.

B-1.2 Ammonium Chloride — solid.

B-1.3 Ammonium Hydroxide Solution — relative density 0·90.

B-1.4 Concentrated Hydrochloric Acid — relative density 1·16.

B-1.5 Dilute Ammonium Nitrate Solution — approximately 2 percent (m/v).

B-2. PROCEDURE

B-2.1 Transfer the aqueous solution reserved in **A-2.1** to a 250-ml graduated flask and make up the volume with water. Pipette out 100 ml of the solution in a beaker and filter, if necessary. Boil the solution or the filtrate with a few drops of concentrated nitric acid and then add 5 g of ammonium chloride. Add ammonium hydroxide solution till the smell of ammonia persists and hydroxides are precipitated. Filter. Dissolve the precipitate in concentrated hydrochloric acid and then add ammonium hydroxide solution. Allow to settle for 20 minutes and then filter. Wash the precipitate with dilute ammonium nitrate solution until free from chlorides. Transfer the filter paper along with the precipitate to a tared crucible (previously heated to constant mass and cooled). Dry in an air-oven, ignite the crucible at about 850°C carefully, cool in a desiccator and weigh. Repeat heating, cooling and weighing till constant mass is obtained.

NOTE — Constant mass may be deemed to have been attained when the change in mass when re-heated and cooled does not exceed 0·000 5 g.

B-3. CALCULATION

B-3.1 Mixed oxides, percent by mass $= \frac{250\,A}{M}$

where

A = mass in g of the ignited precipitate, and
M = mass in g of the material taken for the test in **A-2.1**.

APPENDIX C

[*Table* 1, *Item* (ix)]

DETERMINATION OF CHLORIDES

C-1. APPARATUS

C-1.1 Nessler Cylinders — 50 ml capacity, two (*see* IS : 4161-1967*).

C-2. REAGENTS

C-2.1 Concentrated Nitric Acid — relative density 1·42.

C-2.2 Silver Nitrate Solution — approximately 4 percent (m/v).

C-2.3 Standard Sodium Chloride Solution — Dissolve 0·164 9 g of sodium chloride in water and make up the volume with water to 1 000 ml. Transfer exactly 10 ml of this solution to a 100 ml volumetric flask and dilute to 100 ml. One millilitre of this solution contains 0·01 mg of chloride (as Cl).

C-3. PROCEDURE

C-3.1 Weigh 1·000 g of the material and dissolve in minimum quantity (say about 5 ml) of concentrated nitric acid. Transfer the solution to a 100-ml graduated flask and make up the volume with water to the mark. Transfer 10 ml of this solution to one of the Nessler cylinders, add 1 ml of silver nitrate solution and make up the volume with water to 50 ml. Carry out a control test in the other Nessler cylinder using 4 ml of standard sodium chloride solution in place of the material and the same quantity of other reagents in the same total volume of the reaction mixture. Compare the opalescence produced in the two tubes after 5 minutes.

C-3.2 The limit prescribed in Table 1 shall be taken as not having been exceeded if the opalescence produced in the test with the material is not greater than that produced in the control test.

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*Specification for Nessler cylinders.

APPENDIX D

[*Table* 1, *Item* (x)]

DETERMINATION OF CALCIUM CARBONATE AND MAGNESIUM CARBONATE

D-1. REAGENTS

D-1.1 Standard Hydrochloric Acid — approximately 1 N.

D-1.2 Standard Sodium Hydroxide Solution — approximately 0·1 N.

D-1.3 Methyl Orange Indicator — Dissolve 0·1 g of methyl orange in 100 ml of water.

D-2. PROCEDURE

D-2.1 Weigh accurately about 2 g of the material and dissolve in 50 ml of standard hydrochloric acid and 10 ml of water. Titrate the excess acid with standard sodium hydroxide solution using methyl orange as an indicator.

D-3. CALCULATION

$$\text{Calcium carbonate and magnesium carbonate (as } CaCO_3 \text{), percent by mass} = \frac{5{\cdot}005\,(V_1 N_1 - V_2 N_2)}{M}$$

where

V_1 = volume in ml of standard hydrochloric acid added,

N_1 = normality of standard hydrochloric acid,

V_2 = volume in ml of standard sodium hydroxide solution required for excess acid,

N_2 = normality of standard sodium hydroxide solution, and

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

APPENDIX E

[*Table* 1, *Item* (xi)]

DETERMINATION OF MOISTURE CONTENT

E-0. GENERAL

E-0.1 For the determination of moisture content, Dean and Stark Method has been specified.

E-1. APPARATUS

E-1.1 The Deen and Stark apparatus consists of a glass flask heated by suitable means and provided with a reflux condenser discharging into a trap. The connections between the trap and the condenser, and the flask should be interchangeable ground-glass joints. The trap serves to collect and measure the condensed water, and to return the solvent to the flask. The assembly of apparatus is shown in Fig. 1, and the various components are described below.

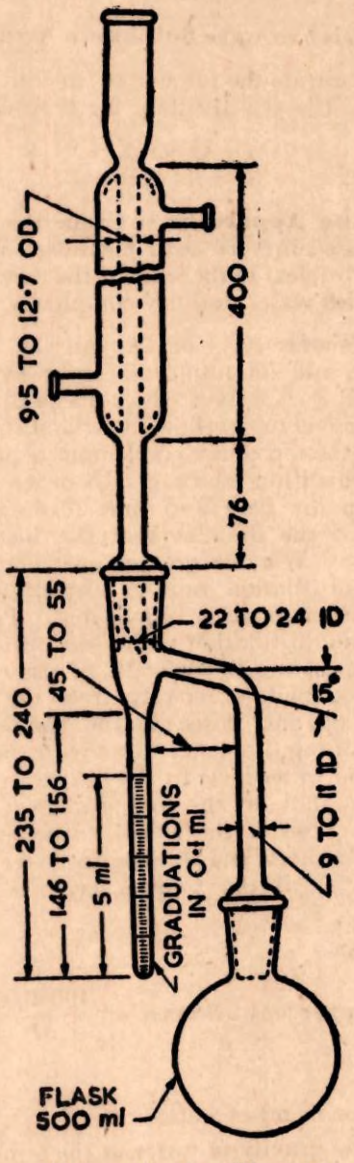
E-1.1.1 *Flask* — of 500 ml capacity and shape shown in Fig. 1, made of hard resistant glass, well-annealed and as free as possible from striae and similar other defects.

E-1.1.2 *Condenser* — a water-cooled, glass reflux condenser, having a jacket approximately 400 mm long and an inner tube of 9·5 to 12·7 mm outside diameter. The tip of the condenser to be inserted in the trap may be ground off at an angle of 30° from the vertical axis of the condenser. When inserted into the trap, the tip of the condenser shall be 6 to 7 mm above the surface of the liquid in the trap after distillation conditions have been established.

E-1.1.3 *Trap* (*or Receiver*) — of 50 ml capacity, made of hard resistant glass, well-annealed and as free as possible from striae and similar other defects, provided with ground-glass joints, with the shape, dimensions and tolerances given in Fig. 1, consisting essentially of the upper chamber, together with the tube and ground joint leading to the flask, and the graduated tube. The graduated portion shall have a capacity of 5 ml when filled to the highest graduation mark. The scale shall cover the range of 0 to 5 ml and shall be graduated at intervals of 0·1 ml. The graduation marks corresponding to each millilitre shall be numbered and carried completely round the tube. The graduation marks midway between the numbered marks shall be carried three-quarters way round the tube and the remaining marks shall be carried half-way round the tube. The error at any indicated capacity shall not exceed 0·05 ml.

E-1.1.4 *Heat Source* — either an oil-bath or an electric heater provided with a sliding rheostat or other means of heat control. The temperature of the oil in the bath should not be very much higher than the boiling point of toluene (*see* **E-2.2**).

E-1.1.5 *Copper Wire* — Long enough to extend through the condenser with one end twisted into a spiral. The diameter of the spiral should be such that it fits snugly within the graduated portion of the receiver and yet can be moved up and down.



All dimensions in millimetres.

FIG. 1 DEAN AND STARK APPARATUS

E-2. REAGENTS

E-2.1 Potassium Dichromate Sulphuric Acid Cleaning Solution

E-2.2 Toluene — Saturate the toluene by shaking with a small quantity of water and distil. Use the distillate for moisture content determination.

E-3. PROCEDURE

E-3.1 Preparing the Apparatus — Clean the entire apparatus with potassium dichromate-sulphuric acid cleaning solution to minimize the adherence of water droplets to the sides of the condenser and the receiver. Rinse thoroughly with water and dry completely before use.

E-3.2 Place approximately 100 g of the material, accurately weighed, in the distillation flask, add 200 ml of toluene and swirl to mix. Assemble the apparatus and fill the receiver with toluene by pouring it through the condenser until it begins to overflow into the distillation flask. Insert a loose cotton plug in the top of the condenser to prevent condensation of atmospheric moisture within the tube. In order that refluxing may be under control, wrap the flask and tube leading to the receiver with asbestos cloth. Heat the flask so that the distillation rate is about 100 drops per minute. When a greater part of the water has distilled over, increase the distillation rate to about 200 drops per minute and continue until no more water is collected. Purge the reflux condenser occasionally during distillation with 5-ml portions of toluene to wash down any moisture adhering to the walls of the condenser. The water in the receiver may be made to separate from the toluene by moving the spiral copper wire up and down in the condenser and the receiver occasionally, thus causing the water to settle at the bottom of the receiver. Reflux until the water level in the receiver remains unchanged for 30 minutes and then shut off the source of heat. Flush the condenser with toluene, making use of the spiral copper wire to discharge any moisture droplets. Immerse the receiver in water at about 27°C for at least 15 minutes or until the toluene layer is clear, and then read the volume of water.

E-4. CALCULATION

E-4.1 Water content, percent by mass $= \dfrac{100\,VD}{M}$

where

V = volume in ml of water,

D = specific gravity of water at the temperature at which th volume of water is read, and

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

E-4.2 Precision — The result of a duplicate determination of moisture content, percent by mass, should not vary by more than ± 0·05 from that of the first determination.

APPENDIX F

(*Clause* 2. 4)

RECIPE FOR COMPOUNDING TEST

F-1. TEST RECIPE

F-1.1 The following test recipe is recommended for checking the performance requirements:

	Parts by Mass
Natural rubber, Grade A	100
Zinc oxide	5
Stearic acid	2
Sulphur	3
Mercaptobenzothiazole	1
Activated calcium carbonate	78

F-2. CURING

F-2.1 Curing shall be done in a press for 45 minutes at 125°C with steam at a pressure of 0·14 MN/m^2 (approx 1·4 kgf/cm^2).

(*Continued from page* 2)

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