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IS 296 (1986): Sodium Carbonate, Anhydrous [CHD 1:
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Indian Standard
SPECIFICATION FOR
SODIUM CARBONATE, ANHYDROUS
(*Third Revision*)

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard
SPECIFICATION FOR
SODIUM CARBONATE, ANHYDROUS
(Third Revision)

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Indian Standard
SPECIFICATION FOR
SODIUM CARBONATE, ANHYDROUS
(*Third Revision*)

0. FOREWORD

0.1 This Indian Standard (Third Revision) was adopted by the Indian Standards Institution on 28 February 1986, after the draft finalized by the Acids, Alkalis and Halides Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard was first formulated in 1951 and subsequently revised in 1965 and 1974. In the present revision, the requirements for the photographic grade have been deleted as the Photographic Sectional Committee, CDC 44, decided to formulate a separate standard for this product exclusively for photographic industry.

0.3 Sodium carbonate, anhydrous, is used in the manufacture of sodium salts and for other special purposes. It is also used as a reagent in analytical chemistry.

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 sodium carbonate, anhydrous.

2. GRADES

2.1 The material shall be of two grades, namely, pure and analytical reagent.

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

3. REQUIREMENTS

3.1 Description — The material shall be in the form of granular or fine white powder, free from foreign matter and visible impurities, and shall be readily soluble in water, forming a clear, colourless solution.

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

3.3 Specific Requirement for Pure Grade Material for the Manufacture of Lead Azide — The pure grade material when used for the manufacture of lead azide shall satisfy all requirements prescribed in col 3 of Table 1, except in respect of its copper content which shall be not more than 0.5 parts per million (as Cu).

4. PACKING AND MARKING

4.1 Packing

4.1.1 The material of the pure grade shall be packed in air-tight containers with polyethylene lining, or as agreed to between the purchaser and the supplier.

4.1.2 The material of the analytical reagent grade shall be packed in glass or other suitable containers on which the material has no action.

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

- a) Name of the material;
- b) Mass and grade of the material in the package;
- c) Name of the manufacturer and recognized trade-mark, if any;
and
- d) Lot or batch number.

4.2.1 Containers for the analytical reagent grade shall be labelled with full analytical data of the characteristics prescribed in col 4 of Table 1.

4.2.2 Containers 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.

TABLE 1 REQUIREMENTS FOR CARBONATE, ANHYDROUS

(Clauses 3.2, 3.3 and 4.2.1)

| Sl. No. | CHARACTERISTIC | REQUIREMENT | | METHOD OF TEST, REF TO CL. NO. IN APPENDIX A |
|---------|---|-------------|--------------------------|--|
| | | Pure Grade | Analytical Reagent Grade | |
| (1) | (2) | (3) | (4) | (5) |
| i) | Loss on ignition at 300°C, percent by mass, <i>Max</i> | 2.0 | 1.0 | A-2 |
| ii) | Total alkalinity (as Na ₂ CO ₃), percent by mass, <i>Min</i> | 99.5 | 99.9 | A-3 |
| iii) | Matter insoluble in water, percent by mass, <i>Max</i> | 0.02 | 0.01 | A-4 |
| iv) | Sulphates (as SO ₄), percent by mass, <i>Max</i> | 0.05 | 0.002 5 | A-5 |
| v) | Chlorides (as Cl), percent by mass, <i>Max</i> | 0.12 | 0.002 | A-6 |
| vi) | Iron (as Fe), percent by mass, <i>Max</i> | 0.004 | 0.000 5 | A-7 |
| vii) | Nitrates (as NO ₃), percent by mass, <i>Max</i> | — | 0.002 | A-8 |
| viii) | Phosphates (as PO ₄), percent by mass, <i>Max</i> | — | 0.001 | A-9 |
| ix) | Silicates (as SiO ₂), percent by mass, <i>Max</i> | — | 0.002 5 | A-10 |
| x) | Heavy metals [as lead (Pb)], parts per million, <i>Max</i> | 10 | 5 | A-11 |
| xi) | Copper (as Cu), percent by mass, <i>Max</i> | 0.003 | — | A-12 |
| xii) | Ammonia (as NH ₄), parts per million, <i>Max</i> | — | 1.0 | A-13 |
| xiii) | Substances reducing iodine (as I), percent by mass, <i>Max</i> | — | 0.005 | A-14 |
| xiv) | Arsenic (as As), parts per million, <i>Max</i> | 3.0 | 0.2 | A-15 |
| xv) | Calcium and magnesium (as Ca and Mg), percent by mass, <i>Max</i> | — | 0.02 | A-16 |
| xvi) | Aluminium (as Al), percent by mass, <i>Max</i> | — | 0.001 | A-17 |
| xvii) | Potassium (as K), percent by mass, <i>Max</i> | — | 0.005 | A-18 |

NOTE — The results shall be reported on as received basis.

5. SAMPLING

5.1 The method of drawing representative test samples of the material and criteria for conformity shall be as prescribed in Appendix B.

APPENDIX A

(Clause 3.2)

ANALYSIS OF SODIUM CARBONATE, ANHYDROUS

A-1. Unless specified otherwise, pure chemicals and distilled water (see IS : 1070-1977*) shall be used in tests.

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

A-2. DETERMINATION OF LOSS ON IGNITION

A-2.1 Procedure — Place about 25 g of the material, accurately weighed, in a porcelain dish, and heat it either in an air-oven or in an electric furnace, or on a sand-bath maintained at 300°C, for one hour. Cool in a desiccator and weigh. Preserve the ignited material for subsequent tests.

A-2.2 Calculation

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

where

M_1 = loss in mass in g, and

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

A-3. DETERMINATION OF TOTAL ALKALINITY

A-3.1 Reagents

A-3.1.1 Standard Hydrochloric Acid — 1 N and 0.1 N.

A-3.1.2 Bromophenol Blue Indicator — Dissolve 0.1 g of bromophenol blue in 100 ml of rectified spirit conforming to IS : 323-1959†.

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

†Specification for rectified spirit (revised).

A-3.2 Procedure — Weigh accurately about 2.75 g of the material obtained in A-2.1 and dissolve in 50 ml of water. Add slowly from a burette 50 ml of standard hydrochloric acid (1 N) and further titrate with standard hydrochloric acid (0.1 N) using bromophenol blue as indicator. Calculate the total volume in terms of hydrochloric acid (1 N).

A-3.3 Calculation

$$\begin{array}{l} \text{Total alkalinity (as Na}_2\text{CO}_3\text{),} \\ \text{percent by mass of ignited material} = \frac{5.3 VN}{M} \end{array}$$

where

V = volume in ml of standard hydrochloric acid (1 N) used in the titration,

N = normality of standard hydrochloric acid, and

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

A-4. DETERMINATION OF MATTER INSOLUBLE IN WATER

A-4.1 Procedure — Weigh accurately 10 to 20 g of the material, transfer it to a 400-ml beaker, add about 200 ml of freshly boiled water, and boil the resulting solution for about 10 minutes. Filter through a tared Gooch crucible or a sintered glass crucible (G No. 4), collecting the filtrate in a 1 000-ml volumetric flask. Transfer the insoluble matter completely to the filter crucible using small volumes of hot water, then wash the crucible 4 times using 5 ml of water for each washing; collect the washings with the filtrate, dry the residue to constant mass at 105 to 110°C. Preserve the solution for subsequent tests.

A-4.2 Calculation

$$\begin{array}{l} \text{Matter insoluble in water,} \\ \text{percent by mass} \end{array} = 100 \times \frac{M_1}{M_2}$$

where

M_1 = mass in g of the residue, and

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

A-5. DETERMINATION OF SULPHATES

A-5.1 For Pure Grade

A-5.1.1 Reagent

A-5.1.1.1 Concentrated hydrochloric acid — See IS : 265-1976*.

*Specification for hydrochloric acid (second revision).

A-5.1.2 Procedure

A-5.1.2.1 Preparation of solution — Weigh accurately 10 g of the material and dissolve in 100 ml of water. Carefully add concentrated hydrochloric acid to make it acidic, and boil to decompose the carbonate. Cool and, if necessary, filter through a folded filter paper. Wash the filter paper thoroughly, collecting the filtrate and washings together.

A-5.1.2.2 Carry out the determination of sulphates with the solution obtained in **A-5.1.2.1** as directed in IS : 2317-1975*.

A-5.2 Analytical Reagent Grade

A-5.2.1 Reagents

A-5.2.1.1 Dilute hydrochloric acid — approximately 2 N.

A-5.2.1.2 Barium chloride solution — approximately 10 percent.

A-5.2.1.3 Methyl orange paper

A-5.2.1.4 Industrial methylated spirit

A-5.2.1.5 Standard sulphate solution — Dissolve 0.148 g of ignited sodium sulphate (Na_2SO_4) in water and dilute to 1 000 ml. One millilitre of this solution contains 0.1 mg of sulphate (as SO_4).

A-5.2.2 Procedure — Dissolve 1 g of the material with 20 ml of water in Nessler cylinder and make slightly acidic to methyl orange paper with hydrochloric acid and add 1 ml in excess. Boil off the carbon dioxide, cool, dilute to 40 ml and add 10 ml of industrial methylated spirit. Mix the contents and add 1 ml of barium chloride solution. Shake vigorously and allow to stand for 1 hour. Carry out a control test in another Nessler cylinder using 0.25 ml of standard sulphate solution and the same quantities of other reagents in the same total volume.

A-5.2.2.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the turbidity or precipitate produced in the test with the material is not greater than that produced in the control test.

A-6. DETERMINATION OF CHLORIDES

A-6.1 For Pure Grade

A-6.1.1 Reagents

A-6.1.1.1 Dilute nitric acid — approximately 4 N and free from chlorides.

*Method for gravimetric determination of sulphates (*first revision*).

A-6.1.1.2 *Calcium carbonate* — precipitated.

A-6.1.1.3 *Potassium chromate indicator* — 5 percent solution.

A-6.1.1.4 *Standard silver nitrate solution* — 0.01 N.

A-6.1.2 Procedure — Dilute the solution reserved in **A-4.1** to 1 000 ml. Transfer 10 ml into a conical flask and add dilute nitric acid in small portions to neutralize the carbonate. Avoid a large excess of the acid. Add precipitated calcium carbonate in small portions until a small amount of it remains after thorough shaking. Add 10 drops of potassium chromate indicator solution. Titrate against standard silver nitrate solution till the reddish brown tinge persists after brisk shaking.

A-6.1.3 Calculation

$$\text{Chlorides (as Cl), percent by mass} = 354.6 \frac{VN}{M}$$

where

V = volume in ml of standard silver nitrate solution used in the titration,

N = normality of standard silver nitrate solution, and

M = mass in g of the material taken for the test in **A-4.1**.

A-6.2 For Analytical Reagent Grade

A-6.2.1 Apparatus

A-6.2.1.1 *Nessler cylinders* — 100-ml capacity.

A-6.2.2 Reagents

A-6.2.2.1 *Dilute nitric acid* — approximately 5 N.

A-6.2.2.2 *Silver nitrate solution* — approximately 5 percent.

A-6.2.2.3 *Standard chloride solution* — Dissolve 0.165 g of sodium chloride, dried at $105 \pm 5^\circ\text{C}$, in water and make up to 1 000 ml. Further dilute 10 ml to 100 ml. One millilitre of the diluted solution contains 0.01 mg of chloride (as Cl).

A-6.2.3 Procedure — Weigh accurately 1.0 g of the material into a Nessler cylinder, dissolve in 40 ml of water and add 12 ml of dilute nitric acid followed by 1 ml of silver nitrate solution. Dilute the contents to the mark with water. Carry out a control test in a similar manner in another Nessler cylinder using 2 ml of standard chloride solution. Compare the turbidity in the two cylinders after 5 minutes.

A-6.2.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the opalescence produced by the material is not greater than that produced in the control test.

A-7. DETERMINATION OF IRON

A-7.1 Apparatus

A-7.1.1 *Nessler Cylinders* — 50-ml capacity.

A-7.2 Reagents

A-7.2.1 *Hydrochloric Acid* — approximately 15 percent (*m/v*).

A-7.2.2 *Ammonium Persulphate*

A-7.2.3 *Potassium Thiocyanate Solution* — approximately 5 percent.

A-7.2.4 *Dilute Sulphuric Acid* — approximately 10 percent (*v/v*).

A-7.2.5 *Standard Iron Solution* — Dissolve 0.702 g of ferrous ammonium sulphate [$\text{FeSO}_4 (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$] in 10 ml of dilute sulphuric acid and dilute with water to 1 000 ml. Dilute 100 ml of the solution again to 1 000 ml. One millilitre of the diluted solution contains 0.01 mg of iron (as Fe).

A-7.3 Procedure — Weigh accurately 1.000 g of the material and dissolve in about 20 ml of water. Add about 5 ml of hydrochloric acid and 30 mg of ammonium persulphate, and boil to oxidize the iron. Cool and transfer to a Nessler cylinder; add 5 ml of potassium thiocyanate solution, dilute with water to the 50-ml mark and stir well. Carry out a control test in another Nessler cylinder, using 4 ml and 0.5 ml of standard iron solution for pure grade and analytical reagent grade, respectively in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture. Compare the colour produced in the cylinders after 5 minutes.

A-7.3.1 The limit prescribed in Table 1 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-8. DETERMINATION OF NITRATES

A-8.1 Reagents

A-8.1.1 *Dilute Sulphuric Acid* — approximately 5 N.

A-8.1.2 *Concentrated Hydrochloric Acid* — same as in **A-5.1.1.1**.

A-8.1.3 Potassium Nitrate Solution — 0.001 N.

A-8.1.4 Concentrated Sulphuric Acid — conforming to IS : 266-1977*.

A-8.1.5 Standard Indigo Carmine Solution — Dissolve 0.20 g of indigo carmine in 400 ml of dilute sulphuric acid, add 20 ml of concentrated hydrochloric acid and sufficient dilute sulphuric acid to produce 1 000 ml. Standardize the solution so that 10 ml added to 3.3 ml of potassium nitrate solution is just decolourized on adding 13 ml of concentrated sulphuric acid and heated to boiling. One millilitre of this solution is equivalent to 0.02 mg of nitrate (as NO_3).

A-8.2 Procedure — Dissolve 1.00 g of the material in 10 ml of dilute sulphuric acid, add 1 ml of standard indigo carmine solution followed by 10 ml of concentrated sulphuric acid and heat to boiling.

A-8.2.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the blue colour produced in the test with the material does not entirely disappear.

A-9. DETERMINATION OF PHOSPHATES

A-9.1 Apparatus

A-9.1.1 Separating Funnels — 200 to 250 ml capacity.

A-9.1.2 pH Meter — with glass electrode.

NOTE — Alternatively, universal standard indicator paper may be used.

A-9.1.3 Nessler Cylinder — 50-ml capacity.

A-9.2 Reagents

A-9.2.1 Dilute Sulphuric Acid — approximately 5 percent (by volume).

A-9.2.2 Ammonium Molybdate — analytical reagent.

A-9.2.3 Concentrated Hydrochloric Acid — same as in A-5.1.1.1.

A-9.2.4 Dilute Hydrochloric Acid — approximately 10 percent (by volume).

A-9.2.5 Ethyl Ether — See IS : 336-1973†.

A-9.2.6 Stannous Chloride Solution — Dissolve 2 g of stannous chloride ($\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$) in concentrated hydrochloric acid and dilute to 100 ml with the acid.

*Specification for sulphuric acid (second revision).

†Specification for ether (second revision).

A-9.2.7 Standard Phosphate Silicate Solution — Dissolve 1.43 g of potassium dihydrogen phosphate (KH_2PO_4) and 23.65 g of sodium silicate ($\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O}$) in water and make up to 1 000 ml. Store this solution in a waxed glass bottle or polyethylene bottle. Dilute 10 ml of this solution to 1 000 ml with water immediately before use. One millilitre of this diluted solution is equivalent to 0.01 mg of phosphate (as PO_4) and to 0.05 mg of silica (as SiO_2).

A-9.3 Procedure — Weigh accurately 1.00 g of the material and dissolve in 50 ml of water in a platinum dish. Digest on the steam-bath for 20 minutes. Cool, neutralize with dilute sulphuric acid to a pH of about 4, and dilute to about 75 ml. Take 1 ml of standard phosphate-silicate solution and make up to about 75 ml. Add 0.5 g of ammonium molybdate to each solution and when it dissolves, adjust the pH to 2 by adding dilute hydrochloric acid. Check the pH with the pH meter or universal pH indicator paper. Heat both the solutions to boiling, cool to room temperature, add 10 ml of concentrated hydrochloric acid to each and dilute to 100 ml with water. Transfer the solutions to separating funnels, add 35 ml of ether to each, shake vigorously and allow to separate. Draw off the aqueous phase and reserve it for determination of silica. Wash the ether phase of each by shaking with 10 ml of dilute hydrochloric acid, allow to separate and drain off and discard this aqueous phase. Add 0.2 ml of freshly prepared stannous chloride solution to each ether extract and shake. If the ether extracts are turbid, wash with 10 ml of dilute hydrochloric acid.

A-9.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of the blue colour produced in the test with the material is not greater than that produced in the control test.

A-10. DETERMINATION OF SILICATES

A-10.1 Apparatus — same as described under A-9.1.

A-10.2 Reagents

A-10.2.1 Concentrated Hydrochloric Acid — same as in A-5.1.1.1.

A-10.2.2 Butanol

A-10.2.3 Dilute Hydrochloric Acid — 1 percent (by volume).

A-10.2.4 Stannous Chloride Solution — same as in A-9.2.6.

A-10.3 Procedure — Add 10 ml of concentrated hydrochloric acid to the solution reserved for the determination of silica (A-9.3) and transfer to separating funnels. Add 40 ml of butanol, shake vigorously, and

allow to separate. Draw off and discard the aqueous phase. Wash the butanol three times with 20-ml portions of dilute hydrochloric acid, discarding the washing each time. Dilute each butanol solution to 50 ml, take 20 ml aliquot of the sample solution and 10 ml aliquot of standard solution and dilute each to 50 ml with butanol. Add 0.5 ml of a freshly prepared 2 percent stannous chloride solution to each and shake. If the butanol extracts are turbid, wash with 10 ml of dilute hydrochloric acid.

A-10.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of the blue colour produced in the test with the material is not greater than that produced in the control test.

A-11. DETERMINATION OF HEAVY METALS

A-11.1 Apparatus

A-11.1.1 *Nessler Cylinders* — 50-ml capacity.

A-11.2 Reagents

A-11.2.1 *Dilute Hydrochloric Acid* — approximately 5 N, and 1 : 99 (*v/v*).

A-11.2.2 *p-Nitrophenol Indicator Solution* — 0.25 percent.

A-11.2.3 *Dilute Ammonium Hydroxide* — 1 : 99 (*v/v*).

A-11.2.4 *Hydrogen Sulphide Solution* — saturated.

A-11.2.5 *Standard Lead Solution* — Weigh accurately 1.60 g of lead nitrate and add 50 ml of nitric acid of analytical reagent grade. Dissolve in water and make up the solution to 1 000-ml mark. From this, pipette out 10 ml of the solution and dilute it with water to 1 000 ml. One millilitre of this solution contains 0.01 mg of lead (as Pb).

A-11.3 Procedure — Dissolve 100 g of the material in 5 ml of dilute hydrochloric acid (5N) and add 20 ml of water. Add 1 drop of *p*-nitrophenol indicator solution and then add, dropwise, dilute ammonium hydroxide until the solution turns yellow. Add dilute hydrochloric acid (1 : 99) dropwise, until the solution becomes colourless and then add 0.5 ml in excess. Transfer to a Nessler cylinder; add 5 ml of hydrogen sulphide solution, dilute to 50 ml mark and shake well. Carry out a control test using 1 ml of standard lead solution for pure grade and 0.5 ml of standard lead solution for analytical reagent grade in place of the material and the same quantities of the other reagents. Compare the colour produced in the two Nessler cylinders.

A-11.3.1 The limit prescribed in Table 1 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. DETERMINATION OF COPPER

A-12.1 Apparatus

A-12.1.1 *Nessler Cylinders* — 100-ml capacity.

A-12.2 Reagents

A-12.2.1 *Copper-Free Water* — Re-distil ordinary distilled water twice in an all-glass apparatus.

A-12.2.2 *Concentrated Sulphuric Acid* — See IS : 266-1977*.

A-12.2.3 *Ammonium Hydroxide* — 20 percent (m/m).

A-12.2.4 *Litmus Paper*

A-12.2.5 *Gum Arabic Solution* — Dissolve 5 g of gum arabic in 100 ml of copper-free water. Filter, if necessary.

A-12.2.6 *Sodium Diethyl Dithio Carbamate Solution* — Dissolve 0.1 g of sodium diethyl dithio carbamate in 100 ml of copper-free water. Filter, if necessary. Preserve in an amber-coloured bottle and store away from light. As the solution deteriorates on storage, it should not be preserved for more than two weeks.

A-12.2.7 *Concentrated Standard Copper Solution* — Dissolve 0.3928 g of copper sulphate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in 250 ml of copper-free water. Pipette out exactly 25 ml of this solution and make up to 100-ml mark. One millilitre of this solution is equivalent to 0.1 mg of copper (as Cu).

A-12.2.8 *Dilute Standard Copper Solution* — Transfer exactly 10 ml of the concentrated standard copper solution and dilute with copper-free water to 100 ml. One millilitre of this solution is equivalent to 0.01 mg of copper (as Cu).

A-12.3 Procedure — Weigh accurately 10.00 g of the material in a 600-ml beaker and dissolve in 50 ml of copper-free water. Add concentrated sulphuric acid (about 8 ml) drop by drop till evolution of carbon dioxide ceases. Place the beaker in a water-bath and evaporate to dryness. Dissolve the sodium sulphate formed in 50 ml of copper-free water and neutralize with ammonium hydroxide, using litmus paper.

*Specification for sulphuric acid (second revision).

Add 2 to 3 ml of excess of ammonium hydroxide and bring the solution to boil. Filter hot to remove all iron which is likely to interfere, if present in large quantities. Transfer the filtered solution to a Nessler cylinder and add 1 ml of gum arabic solution followed by 10 ml each of ammonium hydroxide and sodium diethyl dithio carbamate solution. Carry out a control test using 3 ml of concentrated standard copper solution in the case of the pure grade and 0.5 ml of dilute standard copper solution in the case of the material required for the manufacture of lead azide in place of the material, and the same quantities of other reagents in the same total volume of the reaction mixture. Compare the colour in the two Nessler cylinders after 5 minutes.

A-12.3.1 The limits prescribed for copper (Cu) for the pure grade and for the material required for the manufacture of lead azide shall be taken as not having been exceeded if the colour produced in the test with the material is not deeper than that produced in the appropriate control test.

A-13. DETERMINATION OF AMMONIA

A-13.1 Apparatus

A-13.1.1 *Nessler Cylinders* — 50-ml capacity.

A-13.2 Reagents

A-13.2.1 *Concentrated Hydrochloric Acid* — See IS : 265-1976*.

A-13.2.2 *Sodium Hydroxide Solution* — approximately 5 N.

A-13.2.3 *Potassium Iodide* — crystals.

A-13.2.4 *Mercuric Chloride Solution* — saturated.

A-13.2.5 *Potassium Hydroxide* — solid.

A-13.2.6 *Nessler Solution* — Dissolve 10 g of potassium iodide in 10 ml of ammonia-free water and add to it slowly, with stirring, mercuric chloride solution until a slight permanent precipitate forms. Add 30 g of potassium hydroxide and, when it has dissolved, add 1 ml more of mercuric chloride solution and dilute to 200 ml with ammonia-free water. Allow to settle over-night, decant the clear solution and keep the solution in a bottle closed with a well-fitting rubber stopper.

A-13.2.7 *Standard Ammonia Solution* — Dissolve 2.97 g of ammonium chloride in water and make up to 1 000 ml. Take 10 ml of this and

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

dilute to 1 000 ml. One millilitre of this solution contains 0.01 mg of ammonia (as NH_4).

A-13.3 Procedure — Dissolve 5.000 g of the material in 25 ml of water and add 8.5 ml of concentrated hydrochloric acid. Boil to remove carbon dioxide, cool and transfer to a Nessler cylinder and add 2 ml of sodium hydroxide solution and 2 ml of Nessler solution. Dilute the contents to the mark. Carry out a control test, using 0.5 ml of standard ammonia solution, 2 ml of Nessler solution and 2 ml of sodium hydroxide solution in the same total volume of the reaction mixture.

A-13.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of the colour produced in the test with the material is not greater than that produced in the control test.

A-14. DETERMINATION OF SUBSTANCES REDUCING IODINE

A-14.1 Reagents

A-14.1.1 Starch Solution — Triturate 5 g of starch and 0.01 g of mercuric iodide with 20 ml of water in a mortar. Pour the resulting paste into one litre of boiling water, boil for 3 minutes. Allow the solution to cool and decant off the clear liquid.

A-14.1.2 Standard Iodine Solution — 0.01 N.

A-14.1.3 Dilute Hydrochloric Acid — approximately 5 N.

A-14.2 Procedure — Dissolve 10 g of the material in 100 ml of water. Add 2 ml of starch solution and 20 ml of hydrochloric acid. Titrate the contents with standard iodine solution.

A-14.2.1 The limit prescribed in Table 1 shall be taken to have not been exceeded if not more than 0.4 ml of iodine is required to produce a permanent blue colour.

A-15. DETERMINATION OF ARSENIC

A-15.1 Procedure — Dissolve 1.0 g of the material for pure grade and 5.0 g for analytical reagent grade in 25 ml of water and neutralize with dilute sulphuric acid, using methyl orange as indicator. Carry out the test for arsenic as directed in IS : 2038-1983*, using for comparison standard arsenic trioxide solution containing 0.003 96 g and 0.001 32 g of arsenic trioxide for pure and analytical grades, respectively.

*Method of determination of arsenic (*second revision*).

A-16. DETERMINATION OF CALCIUM AND MAGNESIUM**A-16.1 Reagents**

A-16.1.1 *Dilute Hydrochloric Acid* — approximately 5 N.

A-16.1.2 *Sodium Hydroxide Solution* — approximately 5 N.

A-16.1.3 *Standard Magnesium Solution* — Dissolve 1.013 g of magnesium sulphate ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$) in water and dilute to 1000 ml. One millilitre of the solution contains 0.1 mg of magnesium (as Mg).

A-16.1.4 *Borate Buffer Solution* — Dissolve, in water, 40 g of the sodium borate, 10 g of sodium hydroxide and 5 g of sodium sulphate and dilute the solution to one litre.

A-16.1.5 *Eriochrome Black T Indicator Solution* — Dissolve 0.1 g of eriochrome black T in 20 ml of rectified spirit.

A-16.1.6 *Standard Ethylene Diamine Tetra-Acetate (EDTA) Solution* — Dissolve 40 g of disodium ethylene diamine tetra-acetate dihydrate in water and dilute to one litre. Standardize the solution by titrating against standard magnesium solution following the procedure given in **A-16.2**. Dilute the solution appropriately to obtain N/100 solution.

A-16.2 Procedure — Dissolve 5.00 g of the material in a mixture of 80 ml of water and 20 ml of dilute hydrochloric acid. Boil for 5 minutes. Cool and neutralize to pH 7.0 with sodium hydroxide solution (about 1 ml). Add 1 ml of standard magnesium solution and sufficient borate buffer solution (about 2 ml) to adjust pH to 10.0. Add 0.1 ml of eriochrome black T indicator solution and titrate slowly, while shaking, with standard EDTA solution, using a microburette until a pure blue end point is obtained.

A-16.2.1 The material shall be taken to have passed the test if not more than 4.25 ml of standard EDTA solution is required for the titration.

A-17. DETERMINATION OF ALUMINIUM**A-17.1 Apparatus**

A-17.1.1 *Nessler Cylinders* — 50-ml capacity.

A-17.2 Reagents

A-17.2.1 *Dilute Acetic Acid* — Dissolve 28.5 g of glacial acetic acid in 100 ml of water.

A-17.2.2 Ammonium Aurine Tricarboxylate (Aluminon) Solution — Dissolve 0.1 g of ammonium aurine tricarboxylate in 100 ml of water.

A-17.2.3 Ammonium Carbonate Solution — approximately 5 N.

A-17.2.4 Standard Aluminium Solution — Dissolve 1.680 g of aluminium ammonium sulphate $[\text{AlNH}_4(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}]$ and dilute to 1 000 ml. Further dilute 10 ml of this solution to 100 ml. One millilitre of the diluted solution contains 0.01 mg of aluminium (as Al).

A-17.2.5 Ammonium Acetate — solid.

A-17.3 Procedure — Transfer 10 ml of the sample solution (see A-5.1.2.1) into a Nessler cylinder. Add 10 ml of dilute acetic acid and 1 ml of ammonium aurine tricarboxylate solution. Allow to stand for 5 minutes and then add 10 ml of ammonium carbonate solution. Carry out a control test in another Nessler cylinder, using 1.0 ml of standard aluminium solution, 15 ml of water, 1 g of ammonium acetate, 5 ml of dilute acetic acid, 1 ml of ammonium aurine tricarboxylate solution and adding, after a lapse of 5 minutes, 10 ml of ammonium carbonate solution. Make up the volume in the two cylinders to 50 ml and compare the colour produced in the two cylinders after 5 minutes.

A-17.3.1 The limit prescribed for aluminium shall be taken as not having been exceeded if any pink colour produced in the test with the material is not deeper than that produced in the control test.

A-18. DETERMINATION OF POTASSIUM

A-18.1 Simple Flame Photometer — equipped with interference filter.

A-18.2 Reagents

A-18.2.1 Standard Potassium Chloride Solution — Weigh exactly 1 g of potassium chloride, dissolve in water and dilute to one litre with distilled water in a measuring flask. This solution contains 0.1 g potassium chloride per 100 ml.

A-18.2.2 Calibration Graph — Take 10, 20, 40, 60 and 80 ml of standard potassium solution (see A-18.2.1) and dilute to 100 ml with water in different measuring flasks. Each flask now contains 0.01, 0.02, 0.04, 0.06 and 0.08 g potassium chloride per 100 ml. Use these dilute solutions to obtain a corresponding galvanometer reading as given in the procedure (see A-18.3) and plot the concentration against galvanometer reading in a rectangular coordinate graph. Draw a smooth curve over the points which gives a calibration graph in the range 0.01 to 0.1 percent potassium chloride.

A-18.2.3 Sample Solution — Dissolve approximately 10 g of the sample in minimum quantity of water and dilute to 100 ml in a measuring flask with water.

A-18.3 Procedure

A-18.3.1 Use a flame photometer equipped with atomixer, burner; optical selective device consisting of the reflectors, lenses and diaphragms; and measuring instrument consisting of photocell, amplifier and sensitive galvanometer. The galvanometer scale ranges from 0 to 100 divisions which measures the intensity of the radiation transmitted by the element.

A-18.3.2 Insert the potassium filter corresponding to wave-length 767 nm light burner fed by illuminating gas (laboratory gas) and adjust the specified air pressure between 0.5 and 0.6 kg/cm² and maintain the above air pressure constant such that flame is non-luminous by turning the control knob. First spray water and adjust the pointer to zero in galvanometer scale by zero adjustment knob. Then spray the potassium chloride standard solution (**A-18.2.1**) and adjust the deflection to maximum (100) by using sensitivity control knob. Again spray water to see pointer comes to zero; then spray a standard solution to indicate 100. Repeat till water reads zero and standard solution reads 100 with same adjustment during both the operations. A reading zero by water and with the same adjustment 100 by standard solution indicates that the instrument has been now made ready for measurement.

A-18.3.3 Without altering the earlier adjustment of the instrument spray various diluted solutions prepared in **A-18.2.1** and obtain a calibration graph in the range 0.01 to 0.1 percent potassium chloride. After washing with water, spray the sample solution and obtain the galvanometer reading. From the graph, read out the corresponding concentration of potassium chloride in the solution (say *A*).

A-18.4 Calculation

$$\text{Potassium, percent by mass} = 52.35 \frac{A}{M}$$

where

A = concentration of potassium chloride in the sample solution, and

M = mass in g of dried sample taken for the test.

A P P E N D I X B

(Clause 5.1)

SAMPLING OF SODIUM CARBONATE, ANHYDROUS

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing samples, the following precautions and directions shall be observed.

B-1.1 Samples shall not be taken in an exposed place.

B-1.2 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.3 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.4 The samples shall be placed in clean, dry and air-tight glass or other suitable containers on which the material has no action.

B-1.5 The sample containers shall be of such a size that they are nearly filled by the sample.

B-1.6 Each sample container shall be sealed air-tight after filling, and marked with full details of sampling, the date of sampling and the year of manufacture of the material.

B-2. SCALE OF SAMPLING

B-2.1 Lot — In any consignment of one grade of the material, all the containers of the same size and drawn from a single batch of manufacture shall constitute a lot. If a consignment of one grade of the material is known to consist of different batches of manufacture or of different sizes of containers, then the containers belonging to the same batch and size shall be grouped together and each such group shall constitute a separate lot.

B-2.2 For ascertaining the conformity of the lot to the requirements of the specification, tests shall be carried out for each lot separately. The number of containers (n) to be selected for this purpose shall depend on the size of the lot (N) and shall be in accordance with Table 2.

TABLE 2 SCALE OF SAMPLING*(Clause B-2.2)*

| LOT SIZE | NUMBER OF CONTAINERS TO BE SELECTED |
|---------------|--|
| <i>N</i> | <i>n</i> |
| (1) | (2) |
| Up to 15 | 3 |
| 16 to 40 | 4 |
| 41 to 65 | 5 |
| 66 to 110 | 7 |
| 111 and above | 10 |

B-2.3 These containers shall be selected at random from the lot and in order to ensure randomness of selection, random sampling procedures given in IS : 4905-1968* may be followed.

B-3. TEST SAMPLES AND REFEREE SAMPLE

B-3.1 Preparation of Test Samples

B-3.1.1 Draw with an appropriate sampling instrument a small portion of the material from different parts of each container selected. The total quantity of the material drawn from each container shall be sufficient to conduct the tests for the characteristics given in Table 1 and should be about 400 g.

B-3.1.2 Thoroughly mix all portions of the material drawn from the same container. Out of these portions a small but equal quantity shall be taken from each selected container and shall be well mixed up together so as to form a composite sample weighing not less than 800 g. This composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as referee sample.

B-3.1.3 The remaining portion of the material from each container (after a small quantity needed for the formation of composite sample has been taken) shall be divided into three equal parts, each part weighing not less than 50 g. These parts shall be immediately transferred to thoroughly dried bottles which are then sealed air-tight with stoppers and labelled with all the particulars of sampling given under **B-1.6**. The material in each such sealed bottle shall constitute an individual test sample. These individual samples shall be separated into three

*Methods for random sampling.

identical sets of samples in such a way that each set has an individual test sample representing each container selected. One of these three sets shall be sent to the purchaser, another to the supplier and the third shall be used as a referee sample.

B-3.2 Referee Sample — The referee sample shall consist of the composite sample (*see B-3.1.2*) and a set of individual samples (*see B-3.1.3*) marked for this purpose and shall bear the seals of the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of dispute between the two.

B-4. NUMBER OF TESTS

B-4.1 Tests for the determination of chlorides and iron shall be conducted on each of the individual samples analytical reagent grade.

B-4.2 Tests for all the characteristics for pure grade and analytical reagent grades shall be conducted on the composite sample.

B-5. CRITERIA FOR CONFORMITY

B-5.1 For Individual Samples

B-5.1.1 For Chlorides

B-5.1.1.1 For analytical reagent grade — The lot shall be considered to have passed the requirement if each of the individual samples satisfies the test laid down in **A-6.2.3**.

B-5.1.2 For Iron — The lot shall be considered to have passed the requirement if each of the individual samples satisfied the test laid down in **A-7.3**.

B-5.2 For Composite Sample — The test results on the composite sample shall meet the requirements specified in Table 1.

B-5.2.1 A lot shall be declared as conforming to the specification if it satisfies the requirements for each of the characteristics listed in Table 1.

(Continued from page 2)

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Tata Oil Mills Co Ltd, Bombay
 Mettur Chemical & Industrial Corporation Ltd,
 Mettur Dam

Ballarpur Industries Ltd, New Delhi

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

| QUANTITY | UNIT | SYMBOL |
|---------------------------|----------|--------|
| Length | metre | m |
| Mass | kilogram | kg |
| Time | second | s |
| Electric current | ampere | A |
| Thermodynamic temperature | kelvin | K |
| Luminous intensity | candela | cd |
| Amount of substance | mole | mol |

Supplementary Units

| QUANTITY | UNIT | SYMBOL |
|-------------|-----------|--------|
| Plane angle | radian | rad |
| Solid angle | steradian | sr |

Derived Units

| QUANTITY | UNIT | SYMBOL | DEFINITION |
|----------------------|---------|--------|---|
| Force | newton | N | $1 \text{ N} = 1 \text{ kg}\cdot\text{m}/\text{s}^2$ |
| Energy | joule | J | $1 \text{ J} = 1 \text{ N}\cdot\text{m}$ |
| Power | watt | W | $1 \text{ W} = 1 \text{ J}/\text{s}$ |
| Flux | weber | Wb | $1 \text{ Wb} = 1 \text{ V}\cdot\text{s}$ |
| Flux density | tesla | T | $1 \text{ T} = 1 \text{ Wb}/\text{m}^2$ |
| Frequency | hertz | Hz | $1 \text{ Hz} = 1 \text{ c}/\text{s} (\text{s}^{-1})$ |
| Electric conductance | siemens | S | $1 \text{ S} = 1 \text{ A}/\text{V}$ |
| Electromotive force | volt | V | $1 \text{ V} = 1 \text{ W}/\text{A}$ |
| Pressure, stress | pascal | Pa | $1 \text{ Pa} = 1 \text{ N}/\text{m}^2$ |