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भारतीय मानक
Indian Standard

सोडियम एल्यूमिनेट — विशिष्टि

(दूसरा पुनरीक्षण)

Sodium Aluminate — Specification

(Second Revision)

ICS 71.100.80

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Inorganic Chemicals Sectional Committee, CHD 01

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Inorganic Chemicals Sectional Committee had been approved by the Chemical Division Council.

This standard was originally published in 1967 with a view to helping the producers to manufacture a product of acceptable quality, and in laying down the requirements of the material, due consideration was given to the indigenous production. This standard was revised in 1984 incorporating new limits for iron oxide, silica and moisture as the Committee felt that these elements may affect those products where sodium aluminate is used as a raw material. The methods of estimation for iron oxide and silica have also been modified.

The standard is now being revised to incorporate requirements of cadmium, total chromium, mercury, nickel, and selenium along with their methods of test.

The major application of sodium aluminate is in connection with water treatment and is based on its property of forming flocculant precipitates of aluminium hydroxide which are capable of coagulating, entraining and absorbing finely divided suspended solids and coloured materials. In addition, it possesses the property of coagulating colloidal magnesium hydroxide. Thus, sodium aluminate forms a useful adjunct to the limesoda softening process, particularly when magnesium is also present in water. It is also used in other industries where aluminium is required in an alkaline form or an alkaline coagulant is needed, for example, for printing on fabrics, manufacture of lake colours, sizing of paper, and hardening of building stones, and as filler in soaps. Sodium aluminate is also used in conjunction with sulphate of alumina in the clarification of water supply in water works and in industries using river water. This process is known as double coagulation process.

This standard was formulated by CHD 01 technical committee. The list of experts who had made significant contribution to the formulation of this standard is given at Annex D.

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 ‘Rules for rounding off numerical values (*revised*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

SODIUM ALUMINATE — SPECIFICATION

(Second Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for sodium aluminate.

2 REFERENCES

The Indian Standards given below contain provisions which through reference in this text, constitute provision 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.

IS No.	*Title*
264 : 2005	Nitric acid (*third revision*)
265 : 1993	Hydrochloric acid (*fourth revision*)
299 : 2012	Alumino-ferric — Specification (*fifth revision*)
1070 : 1992	Reagent grade water (*third revision*)
2088 : 1983	Methods for determination of arsenic (*second revision*)
3025	Methods of sampling and test (physical and chemical) for water and wastewater
(Part 28) : 1986	Sulphite (*first* revision)
(Part 41) : 1992	Cadmium (*first revision*)
(Part 54) : 2003	Nickel (*first revision*)
8883 (Part 1) : 2005	Methods of sampling chemical and chemical products: Part 1 General requirements and precautions (*first revision*)
9506 : 1980	Method for determination of sulphate (volumetric)

3 REQUIREMENTS

3.1 Description

The material shall be a white granular mass. It absorbs moisture and carbon dioxide rapidly when exposed to atmosphere and results in caking.

3.2 Stability

Ten grams of the material when dissolved in 100 ml of freshly distilled water and allowed to stand for 24 h in a closed bottle, shall not show appreciably larger flocculation or precipitation than that present in the beginning.

3.3 The material shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in Annex A. Reference to the relevant clauses of Annex A is given in column 4 of the Table 1.

4 PACKING AND MARKING

4.1 Packing

The material shall be suitably packed in mild steel black plate drums with polyethylene liner or as agreed to between the purchaser and the supplier. As the material has a tendency to absorb moisture and carbon dioxide from the atmosphere which results in caking, the exposure of the material direct to air shall be avoided.

4.2 Marking

The packages shall be securely closed and bear legibly and indelibly the following information:

a) Name of the material;
b) Name of the manufacturer and his recognized trade-mark, if any;
c) Batch number;
d) Gross and net mass; and
e) Date of manufacture.

4.3 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

5 SAMPLING

The procedure for drawing representative samples of the material, the number of tests to be made on the samples and the criteria for conformity of the lot to the requirements of this specification shall be as given in Annex C.

Table 1 Requirements for Sodium Aluminate

(*Clause* 3.3)

Sl No. (1)	**Characteristics** (2)	**Requirements** (3)	**Method of Test** (Ref to Clause in Annex) (4)
i)	Caustic alkalinity (as Na_2O), percent by mass, *Min*	40.0	A-3
ii)	Water-soluble aluminate (as Al_2O_3), percent by mass, *Min*	52.0	A-4
iii)	Iron oxide (as Fe_2O_3), parts per million, *Max*	100	A-5
iv)	Matter insoluble in water, percent by mass, *Max*	1.2	A-6
v)	Silica (as SiO_2), parts per million, *Max*	200	A-7
vi)	Sulphates (as Na_2SO_4), percent by mass, *Max*	0.2	A-8
vii)	Chlorides (as NaCl), percent by mass, *Max*	0.2	A-9
viii)	Carbonates (as Na_2CO_3), percent by mass, Max	1·6	A-10
ix)	Lead (as Pb), parts per million, *Max*	20	A-11
x)	Arsenic (as As_2O_3), parts per million, *Max*	20	A-12
xi)	Cadmium (as Cd), ppm, *Max*	6	A-13
xii)	Total chromium (as Cr), ppm, *Max*	100	A-14
xiii)	Mercury (as Hg), ppm, *Max*	2	A-15
xiv)	Nickel (as Ni), ppm, *Max*	40	A-16
xv)	Selenium (as Se), ppm, *Max*	20	A-17
xvi)	Moisture content, percent by mass, *Max*	4.0	Annex B

ANNEX A

(*Clause* 3.3, *Table* 1)

METHODS OF TEST FOR SODIUM ALUMINATE

A-1 QUALITY OF REAGENTS

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

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

A-2 PREPARED SOLUTION

Dissolve about 5 g of the accurately weighed material in carbon dioxide-free water. Make up the volume to 500 ml in a volumetric flask and filter the solution. This solution shall be the prepared solution to be used in tests.

A-3 DETERMINATION OF CAUSTIC ALKALINITY (AS Na_2O)

A-3.1 Reagents

A-3.1.1 *Standard Hydrochloric Acid* — 0.5 N.

A-3.1.2 *Phenolphthalein Indicator* — Dissolve 0·1 g in 100 ml of 60 percent rectified spirit**.**

A-3.1.3 *Barium Chloride Solution* — Approximately 10 percent (*m*/*v*).

A-3.2 Procedure

Take 100 ml of the prepared solution in a conical flask and add 20 ml of barium chloride (neutralized) solution. Titrate it against standard hydrochloric acid using phenolphthalein as indicator.

A-3.3 Calculation

Caustic alkalinity (as Na_2O), percent by mass =

$$\frac{3.10 \times V \times N}{M}$$

Where,

V = volume in ml, of standard hydrochloric acid required for titration;

N = normality of standard hydrochloric acid; and

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

A-4 DETERMINATION OF WATER-SOLUBLE ALUMINATE (AS Al_2O_3)

A-4.1 Principle

Water-soluble aluminates are determined by precipitating the material with ammonium hydroxide twice, igniting and weighing as R_2O_3. The mass of oxides due to iron and titanium is deducted from it to calculate the mass of Al_2O_3.

A-4.2 Reagents

A-4.2.1 *Ammonium Chloride* — Solids.

A-4.2.2 *Concentrated Nitric Acid* — *See* IS 264.

A-4.2.3 *Dilute Ammonium Hydroxide* — Approximately 9 N.

A-4.2.4 *Dilute Hydrochloric Acid* — 1 : 4 (*v*/*v*)

A-4.2.5 *Ammonium Nitrate Solution* —Approximately 2 percent (made neutral with ammonium hydroxide to methyl red or phenol red).

A-4.2.6 *Methyl Red Indicator Solution* — 0.15 g in 500 ml of water.

A-4.3 Procedure

Take 25 ml of the prepared solution and dilute to 100 ml. Add 5 g of ammonium chloride and 3 to 4 ml of nitric acid. Boil the solution for a few minutes. Add 2 to 3 drops of methyl red solution. Add ammonium hydroxide dropwise, with stirring, until the colour of the solution changes to distinct yellow. Boil for one minute, filter through a filter paper (Whatman No. 41 or its equivalent) and wash the precipitate three times with hot ammonium nitrate solution. Transfer the precipitate to the original beaker washing the filter paper with the dilute hydrochloric acid.

A-4.3.1 Add hot dilute hydrochloric acid sufficient to dissolve the precipitate and boil. Add methyl red indicator and make it faintly ammoniacal as before. Boil again for one minute and filter the precipitate as before. Wash the precipitate with hot ammonium nitrate solution until free from chlorides and transfer it with the filter paper to a crucible, previously ignited to constant mass. Dry and ignite at a low temperature until all carbon is burnt off; then complete the ignition at 1 100°C for one hour. Cool the residue in a desiccator over a good desiccant and weigh. Re-ignite it until constant mass is attained.

A-4.3.2 Determine the percentage of aluminium oxide by deducting from it the percentage of iron oxide as obtained under **A-5**.

A-5 DETERMINATION OF IRON OXIDE (AS Fe_2O_3)

A-5.1 Principle

Iron oxide is determined calorimetrically using o-phenanthroline. 1,10-phenanthroline is a ligand and binds to Fe^{+2}. This complex is capable of absorbing radiation in the visible range and thus its concentration can be determined using spectrophotometer/ calorimeter.

A-5.2 Reagents

A-5.2.1 *Standard Iron Solution (10 µg/ml)* — Dissolve 0.491 1 g of ferrous ammonium sulphate [($FeSO_4$). (NH_4)2 SO_4. $6H_2O$)] in water. Add 10 ml of 1 : 1 sulphuric acid and dilute with water to 1 000 ml. Pipette out 50 ml of the solution and dilute with water to 500 ml. One millilitre of this solution is equivalent to 0.01 mg of iron (as Fe_2O_3).

A-5.2.2 *Sulphuric Acid* — 1 : 1.

A-5.2.3 *Hydroxylamine Hydrochloride Solution* — 10 percent (*m/v*).

A-5.2.4 *o-Phenanthroline Solution* — Dissolve 0.1 g of *o*-phenanthroline in 2 ml of methanol and dilute to 100 ml with water and store in a dark bottle with a ground-glass stopper.

A-5.2.5 *Sodium Acetate Solution* — 2 M.

A-5.3 Procedure

Take 10, 20, 30, 40 and 50 ml aliquots of standard iron solution in separate 100 ml beakers. Adjust the *p*H to 2 to 3 using 1 : 1 sulphuric acid and then between 3 to 4 using sodium acetate solution. Add 2 ml of hydroxylamine hydrochloride solution and 5 ml of o-phenanthroline solution. Dilute with water the contents of each beaker to 100 ml. Determine the absorbance after 30 min against reagent blank at 510 nm using a spectrophotometer. Plot a calibration curve of absorbance against concentration of 1.0 µg/m1 to 5 µg/m1 as obtained by following the above dilution steps using the standard iron solution.

A-5.3.1 Dissolve in water 2 g of the material, accurately weighed, and dilute with water to 100 ml. Take 10 ml aliquot in a 100 ml beaker. Adjust the *p*H to 2 to 3 using 1 : 1 sulphuric acid and then between 3 to 4 using sodium acetate solution. Add 2 ml of hydroxylainine hydrochloride solution and 5 ml of o-phenanthroline solution. Dilute with water the contents of the beaker to 100 ml. Determine the absorbance after 30 min against reagent blank at 510 nm using a spectrophotometer. Read the concentration for the absorbance determined using the calibration curve.

A-5.4 Calculation

$$\text{Iron oxide (as } Fe_2O_3 \text{), percent by mass} = \mu\text{g/ml of } Fe_2O_3 \text{ from the calibration curve} \times 0.05$$

A-6 DETERMINATION OF MATTER INSOLUBLE IN WATER

A-6.1 Procedure

Weigh accurately 10 g of the material and dissolve by stirring in 100 ml of water. Filter through a sintered glass crucible. Wash the residue with hot water till it is free from alkali. Dry the residue at 13°C ± 2°C to constant mass.

A-6.2 Calculation

$$\text{Matter insoluble in water, percent by mass} = \frac{100 \times m}{M}$$

where,

m = Mass in g, of the dried residue; and

M = Mass in g, of the material taken for the test.

A-7 DETERMINATION OF SILICA (AS SIO_2)

A-7.1 Principle

Silica is determined calorimetrically using ammonium molybdate.

A-7.2 Reagents

A-7.2.1 *Standard Silica Solution (10 µg/ml)* — Dissolve 0.314 g of dried sodium hexafluorosilicate (Na_2SiF_6) in hot water and dilute to 500 ml. Pipette out 50 ml aliquot and dilute with water to 1 000 ml. One millilitre of this solution is equivalent to 0.01 mg of silica (as SiO_2).

A-7.2.2 *Sulphuric Acid* — 1 : 1 and 10 M.

A-7.2.3 *Ammonium Molybdate Solution* — 10 percent (*m/v*).

A-7.2.4 *Reducing Agent* — 0.7 g of sodium sulphite and 0.15 g of 1-amino-2 naphthol-4 sulphonic acid in 10 ml of water, dissolved in 9 g of sodium metabisulphite in 70 ml of water.

A-7.2.5 *Boric Acid* — 5 percent (m/v).

A-7.2.6 *Hydrofluoric Acid* — 45 percent.

A-7.3 Procedure

Take 10, 20, 30, 40 and 50 ml aliquots of standard silica solution in separate 500 ml plastic beakers. Adjust the *p*H to 2.0 with 1 : 1 sulphuric acid. Add 50 ml of boric acid followed by exact addition to 4 drops of hydrofluoric acid. Add again 50 ml of' boric acid and keep in a constant temperature bath at 40°C for 20 min. Add 4 ml of ammonium molybdate solution and keep at 48°C for further 20 min. Remove the beakers and add 20 ml of 10 M sulphuric acid and 5 ml of reducing agent. Dilute with water the contents of each beaker to 200 ml. Determine the absorbance after 15 min against reagent blank at 650 nm using a spectrophotometer. Plot a calibration curve of absorbance against concentration of 0.5 µg/ml to 2.5 µg/ml as obtained by following the above dilution steps using the standard silica solution.

A-7.3.1 Dissolve in water 0.5 g of the material, accurately weighed, and dilute with water to 500 ml. Take 10 ml aliquot and dilute with water to 100 ml. Further, take 20 ml of this aliquot in a beaker and adjust the *p*H to 2.0 with 1 : 1 sulphuric acid. Transfer the contents to a 500 ml plastic beaker. Add 50 ml of boric acid followed by exact addition of 4 drops of hydrofluoric acid. Add again 50 ml of boric acid and keep in a constant temperature bath at 40°C for 20 min. Add 4 ml of ammonium molybdate solution and keep at 40°C for further 20 min. Remove the beaker and add 20 ml of 10 M sulphuric acid and 5 ml of reducing agent. Dilute with water the contents of the beaker to 200 ml. Determine the absorbance after 15 min against reagent blank at 650 nm using a spectrophotometer. Read the concentration for the absorbance determined using the calibration curve.

A-7.4 Calculation

$$\text{Silica (as SiO}_2\text{), percent by mass} = \mu\text{g/ml of SiO}_2 \text{ from the calibration curve} \times 10$$

A-8 DETERMINATION OF SULPHATES (AS NA_2SO_4)

A-8.1 Principle

Sulphates are determined by precipitation with barium chloride solution.

A-8.2 Reagents

A-8.2.1 *Barium Chloride Solution* — Approximately 10 percent.

A-8.2.2 *Concentrated Hydrochloric Acid* — *see* IS 265.

A-8.3 Procedure

Take 50 ml of the prepared solution in a beaker. Acidify with concentrated hydrochloric acid so that all the aluminium hydroxide precipitates dissolve and add, with stirring, 10 ml of hot barium chloride solution slowly. Boil gently for 5 min and keep it for 2 h on a steam bath. Allow the contents of the beaker to stand for 4 h and filter through a filter paper (Whatman No. 42 or its equivalent). Wash the precipitates with hot water till it is free from chlorides. Transfer the residue in a porcelain crucible and ignite it again at 800°C, cool and weigh till constant mass is obtained.

A-8.3.1 As the maximum limit for lead is 20 parts per million, no correction is necessary for lead content:

A-8.4 Calculation

$$\text{Sulphates (as Na}_2\text{SO}_4\text{), percent by mass} = \frac{60.86 \times m}{M}$$

where,

m = mass in g, of the residue of barium sulphate obtained; and

M = mass in g, of the material taken for the test (*see* **A-7.2**).

A-8.5 Alternatively, the sulphate content may also be estimated in accordance with **3** of IS 9506.

A-9 DETERMINATION OF CHLORIDES (AS NaC1)

A-9.1 Principle

Chlorides are determined by titration with silver nitrate solution using potassium chromate as indicator. When the entire chloride is precipitated as silver chloride, chromate ions combine with silver to form red silver chromate.

A-9.2 Reagents

A.9.2.1 Dilute Nitric Acid — Approximately 5 N.

A-9.2.2 Sodium Bicarbonate — Solid.

A-9.2.3 Potassium Chromate Solution — Approximately 5 percent (m/v).

A-9.2.4 Standard Silver Nitrate Solution — 0.1 N.

A-9.3 Procedure

Take 25 ml of the prepared solution in a 250m1 conical flask. Just acidify with dilute nitric acid. Neutralize the excess of the acid by the addition of sodium bicarbonate. Add 1 ml of potassium chromate solution and titrate with standard silver nitrate solution with vigorous shaking until the first permanent colour deviation from the pure yellow of the suspension is obtained. Carry out a blank titration, using the same quantities of other reagents in the same total volume of the reaction mixture.

A-9.4 Calculation

$$\text{Chlorides (as NaCl), percent by mass} = \frac{5.846 \times N\,(V_1 - V_2)}{M}$$

where,

N = normality of the standard silver nitrate solution;

V_1 = volume in ml, of standard silver nitrate solution required for titration;

V_2 = volume in ml, of standard silver nitrate solution required for blank; and

M = mass in g, of the material contained in the aliquot.

A-10 DETERMINATION OF CARBONATES (AS Na_2CO_3)

A-10.1 Principle

Carbon dioxide is generated by adding dilute hydrochloric acid in vacuum and is absorbed in an excess of alkali which is back titrated with a standard hydrochloric acid.

A-10.2 Apparatus

A-10.2.1 *Kjeldahl Flask* — 300 ml capacity.

A-10 2.2 *Drechsel Bottle* — Provided with two taps.

A-10.2.3 *Vacuum Pump* — To give pressure less than 10 cm of mercury.

A-10.2.4 *Tap Funnel*

A-10.3 Reagents

A-10.3.1 *Methyl Orange Indicator* — Dissolve 0.01 g of methyl orange in 100 ml of water.

A-10.3.2 *Sodium Hydroxide Solution* — 1 N.

A.10.3.3 *Barium Chloride Solution* — 10 percent.

A-10.3.4 *Phenolphthalein Indicator* — Dissolve 0·1 g of phenolphthalein in 100 ml of 60 percent rectified spirit.

A-10.3.5 *Dilute Hydrochloric Acid* — 5 N, 1 N and 0·1 N.

A-10.4 Procedure

Take 5 g of the accurately weighed material in a 300 ml Kjeldahl flask A and add about 100 ml of water and two drops of methyl orange indicator. Assemble the apparatus as shown in Fig. 1. Provide the flask A with a rubber bung through which passes a short bend of wide capillary tubing and a tap funnel B reaching nearly to the bottom of the flask. Place 20 ml of sodium hydroxide solution and 1 ml of phenolphthalein indicator in a Drechsel bottle C provided with taps E and F. Connect this bottle to the Kjeldahl flask and evacuate the whole assembly to less than 10 cm mercury pressure using a vacuum pump. Introduce 40 ml of 5 N hydrochloric acid through the tap funnel into the flask (the precipitated aluminium hydroxide should redissolve and excess acid should be present as indicated by methyl orange), avoiding the admission of air. Keep about 1 ml of acid in the funnel to prevent air leaking during subsequent operation. Heat the flask over a small flame until the outlet capillary tube becomes hot, shaking the Drechsel bottle occasionally to absorb the carbon dioxide. The solution in Drechsel bottle should remain pink. Fill the flask A with boiling water through the tap to flush out any traces of carbon dioxide held back in the flask. Shake the Drechsel bottle and disconnect it after removing the vacuum by opening the outlet tap X. Titrate the excess sodium hydroxide with 1 N hydrochloric acid (phenolphthalein already present) so as not to overstep the end point. Boil off carbon dioxide. It is better to complete this titration near the end point with 0.1 N hydrochloric acid till the solution is just colourless. Add two drops of methyl orange indicator and excess of 1 N hydrochloric acid. Shake well and titrate with sodium hydroxide solution. Carry out the blank in the same way.

A-10.5 Calculation

Carbonates (as Na_2CO_3), percent by mass =

$$\frac{V_1 - (V_3 - V_2) \times 0.053 \times 100}{M}$$

where,

V_1 = excess of standard hydrochloric acid (1 N) added to the solution,

V_2 = volume of standard sodium hydroxide solution consumed in the titration,

V_3 = volume of standard hydrochloric acid (1 N) used for blank titration, and

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

A-11 DETERMINATION OF LEAD (AS Pb)

A-11.1 Principle

Lead is determined by comparing the colour produced by the material with hydrogen sulphide solution against that produced by a standard lead solution.

A-11.2 Apparatus

A-11.2.1 *Nessler Cylinders* — 50 ml capacity.

A-11.3 Reagents

A-11.3.1 *Concentrated Hydrochloric Acid — See* IS 265.

A-11.3.2 *Standard Lead Solution* — Dissolve 1.60 g of lead nitrate in water and make up the volume to 1 000 ml. Transfer exactly 10 ml of the solution to a volumetric flask and dilute it again with water to 1 000 ml mark. One millilitre of this solution contains 0.01 mg of lead (as Pb). The dilute solution shall be freshly prepared.

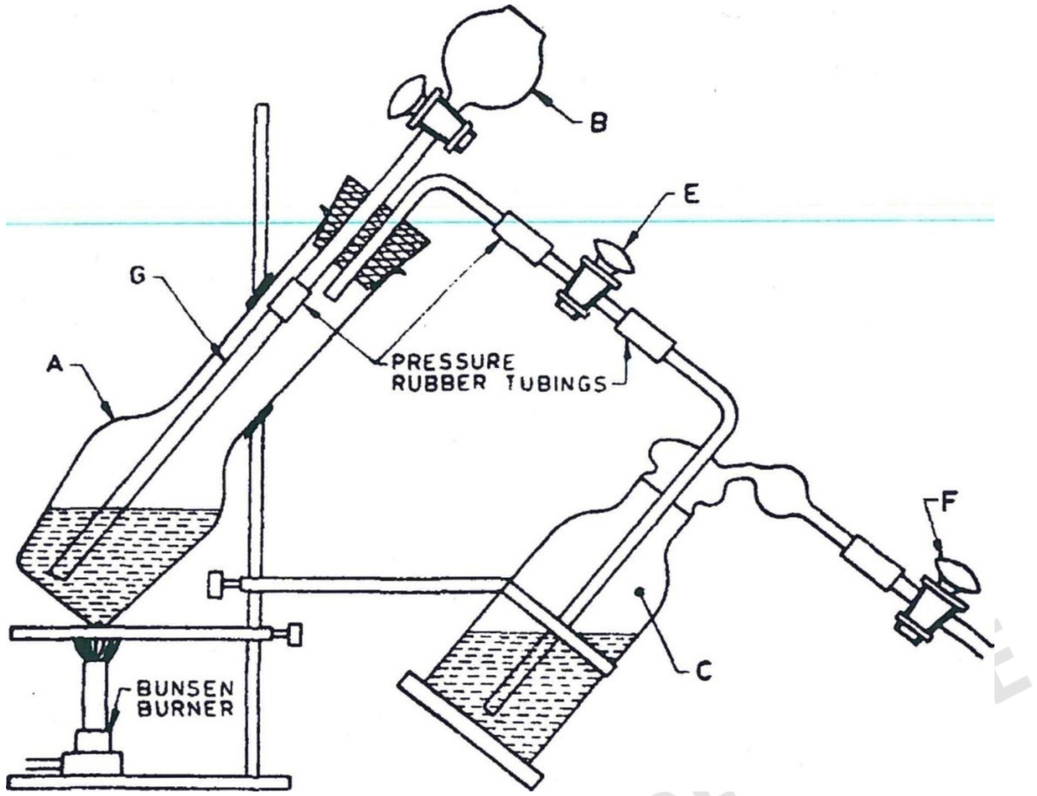

FIG. 1 APPARATUS FOR THE DETERMINATION OF CARBONATES

A-11.3.3 *Acetic Acid* — Approximately 1 N.

A-11.3.4 *Hydrogen Sulphide Solution* — Saturated.

A-11.4 Procedure

Weigh 1.0 g of the material (2.5 g in case of water treatment meant for drinking purpose) and add 15 ml of water. Add 6 ml of concentrated hydrochloric acid and evaporate to dryness on a water-bath. Dissolve the residue in water and transfer this solution to a Nessler cylinder. Dilute it with water to 30 ml and add 1 ml of acetic acid and 10 ml of hydrogen sulphide solution. In the second Nessler cylinder, carry out a control test using 5 ml of standard lead solution and the same quantities of other reagents. Dilute the contents of each cylinder to 50 ml and shake well. Compare the colour produced in the two cylinders.

A-11.4.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced with the material is not greater than that produced in the control test.

A-11.5 Spectrophotometric Method

A-11.5.1 *Principle* — An acidified sample containing microgram quantities of lead is extracted with dithizone solution in chloroform. The extraction is carried out in the presence of strong ammonical citrate-cyanide reducing agent (*p*H 10 to 11.5). The quantity of lead present in the sample is determined spcctrophotometrically by measuring the absorbance of the chloroform extract at 510 nm containing the lead dithizonate complex.

A-11.5.2 *Minimum Detection Limit* — 1.0 µg Pb/10 ml dithizone solution (extract).

A-11.5.3 *Apparatus*

A-11.5.3.1 *Spectrophotometer,* for use at 510 nm with a path length of 1 cm or longer.

A-11.5.3.2 *p*H *meter*

A-11.5.3.3 *Standard volumetric glasswares*

A-11.5.3.4 *TEF beaker,* 100 ml for acid digestion.

A-11.5.3.5 *Separatory funnels,* 250 ml and 500 ml.

A-11.5.3.6 All glasswares are to be cleaned with 1 : 1 HNO_3, and rinsed thoroughly with distilled water.

A-11.5.4 *Reagents*

A-11.5.4.1 *Quality of reagents* — Only analytical or equivalent grade reagents, unless specified otherwise, are to be used. All reagents are to be prepared in lead-free distilled water.

A-11.5.4.2 *Stock lead solution* — Dissolve 0.1599 g lead nitrate [$(Pb(NO_3)_2$, minimum purity, 99.5 percent (*w/w*)] in about 200 ml of water. Add 10 ml concentrated HNO_3 and dilute to 1 000 ml with water, 1.0 ml of this solution will contain 100 µg of Pb.

A-11.5.4.3 *Standard lead solution* — Dilute 2.0 ml of stock lead solution to 100 ml with water, 1.0 ml of this solution will contain 2 µg of Pb.

A-11.5.4.3.1 *Nitric acid* — Concentrated (18N).

A-11.5.4.3.2 *Nitric acid — Dilute* —20 percent, *v/v*.

A-11.5.4.3.3 *Ammonium hydroxide* — Concentrated (14N).

A-11.5.4.3.4 *Ammonium hydroxide* — Dilute 10 percent, *v/v* and 1 percent, *v/v*.

A-11.5.4.3.5 *Citrate-cyanide reducing solution* — Dissolve 200 g anhydrous ammonium citrate [$(NH_4)_2 HC_6H_5O_7$] 10 g anhydrous sodium sulphite (Na_2SO_3), 5 g hydroxylamine hydrochloride, 20 g potassium cyanide (KCN) in water and dilute to 500 ml, and mix with one litre of concentrated NH_4OH.

NOTE — KCN is a poisonous solution. Handle with extreme care and do not pipette by mouth.

A-11.5.5 *Stock Dithizone Solution*

A-11.5.5.1 Dissolve 25 mg dithizone in about 50 ml chloroform ($CHCl_3$) taken in a 200 ml beaker and filter through Whatman No. 42 (or equivalent) filter paper. Collect the filtrate and two washings (10 ml each) in a 250 ml conical flask. Transfer the combined filtrate to a 500 ml separatory funnel. Add about 100 ml 1 percent (*v/v*) NH_4OH solution, shake moderately for about 1 min. Transfer the $CHCl_3$ layer to another 250 ml separatory funnel retaining the orange-red aqueous layer in the 500 ml separatory funnel. Repeat the extraction (of the $CHCl_3$ layer) with 100 ml of 1 percent (*v/v*) NH_4OH solution, transfer the $CHCl_3$ layer to another 250 ml separatory funnel and the aqueous layer to the original 500 ml separatory funnel containing the first extract. One more repetition, of extraction and transferring to the main aqueous layer is carried out. To the combined aqueous extract in the 500 ml separatory funnel add 1:1 HC1 in 2 ml portions, mixing after each addition, until dithizone precipitation is complete and the solution is no longer orange-red. Extract the precipitated dithizone with three 25 ml portions of $CHCl_3$. Dilute the combined extract to 250 ml with $CHCl_3$, 1 ml of this solution will contain 100 µg of dithizone.

A-11.5.5.2 *Working dithizone solution* — Dilute 100 ml stock dithizone solution to 250 ml in a standard volumetric flask with $CHCl_3$, 1 ml of this solution will contain 40 µg of dithizone.

A-11.5.6 *Procedure*

A-11.5.6.1 *Sample digestion*

Digest all samples for lead as per standard digestion procedure using $HNO_3H_2SO_4$ and HNO_3-$HClO_4$. To 100 ml acidified sample (*p*H = 2) add 20 ml of dilute (20 percent, *v/v*) HNO_3, filter if required through a filter paper (Whatman No. 41 or equivalent), and transfer it to a 250 ml separatory funnel. Add 60 ml ammoniacal citrate-cyanide solution, mix and cool to room temperature. Add 10 ml of dithizone working solution. Shake the stoppered funnel vigorously for about 30 s, allow to stand (to get two separate layers). Discard 1-2 ml $CHCl_3$ layer and then fill the absorption cell. Measure the absorbance at 510 nm using working dithizone solution as reagent blank.

A-11.5.7 *Calibration Curve*

Plot a calibration curve using at least five standard lead solutions, after adding 50 ml ammoniacal citrate-cyanide solution to the individual lead standard solutions and extracting the same with 10 ml of dithizone working solution.

A-11.5.8 *Calculation*

mg Pb/litre = µg (in 10 ml extract obtained from calibration curve)/Volume of sample (ml)

A-11.5.9 Alternatively, it can also be analyzed by ICP-OES. AAS method as prescribed in IS 12074 shall be the referee method.

A-12 DETERMINATION OF ARSENIC (AS As_2O_3)

A-12.1 Procedure

Dissolve 0.3 g of material in the minimum amount of water. Carry out the test for arsenic as prescribed in IS 2088, using for comparison a stain obtained with 0.008 mg of arsenic trioxide (as As_2O_3).

A-12.2 The limit prescribed in Table 1 shall be taken as not having been exceeded if the length and intensity of the stain is not greater than that produced in the control sample.

A-12.3 Atomic Absorption Spectrophotometer (AAS) as prescribed in IS 11124 shall be the referee method. Alternately, it can be also analyzed by ICP-OES.

A-13 DETERMINATION OF CADMIUM

Follow the method by AAS (referee method) as prescribed in IS 3025 (Part 41). Alternatively it can be also analyzed by ICP-OES.

A-14 DETERMINATION OF TOTAL CHROMIUM

Follow the method as given in **A-12** of IS 299, but using 0.2 g of sample in water (instead of 1 g) as per procedure laid in **A-12.3** of IS 299.

A-15 DETERMINATION OF MERCURY

Follow the method as given in **A-10** of IS 299, but weighing 1 g of sample in water (instead of 5 g) as per procedure laid in **A-10.5.2** of IS 299.

A-16 DETERMINATION OF NICKEL

Follow the method by AAS (referee method) as prescribed in IS 3025 (Part 54). Alternatively it can be also analyzed by ICP-OES.

A-17 DETERMINATION OF SELENIUM

Follow the method by AAS (referee method) as prescribed in IS 3025 (Part 28). Alternatively it can be also analyzed by ICP-OES.

ANNEX B

(*Table* 1)

B-1 DETERMINATION OF MOISTURE

B-1.1 Procedure

Take 5 g of the accurately weighed material in a tared squat type bottle and dry it at 130°C ± 2°C in an oven till constant mass is obtained. This temperature has been specified in order to reduce the time of the heating.

B-1.2 Calculation

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

Where,

M_1 = mass in g, of material taken; and

M_2 = mass in g, of the material after heating in the oven.

ANNEX C

(*Clause* 4.1)

SAMPLING OF SODIUM ALUMINATE

C-1 GENERAL REQUIREMENTS OF SAMPLING

Reference for general requirements of sampling, reference may be made to IS 8883 (Part 1).

C-2 SCALE OF SAMPLING

C-2.1 Lot

In any consignment, all the containers of the same size and drawn from the same batch of manufacture shall be grouped together to constitute a lot.

C-2.2 For ascertaining the conformity of the material in the lot to requirements of this specification, tests shall be carried out for each lot separately. The number of containers to be selected for this purpose shall depend upon the size of the lot and shall be in accordance with Table 2.

Table 2 Number of Containers to be Selected for Sampling

(*Clause* C-2.2)

Sl No. (1)	Number of Containers in the Lot N (2)	Number of Containers to be Selected n (3)
i)	1 to 50	3
ii)	51 to 100	4
iii)	101 to 300	5
iv)	301 and above	7

C-2.3 The containers shall be selected at random from the lot. To ensure randomness of selection, a random number table may be used. In case such a table is not available, the following procedure may be adopted:

> Starting from any container in the lot, count them as 1, 2,3,……. up to r and so on in one order. Every rth container thus counted shall be withdrawn to constitute the sample, where r is the integral part of N/n, N being the number of containers in the lot and n the number of containers to be selected.

C-3 PREPARATION OF SAMPLES

C-3.1 From each of the selected containers draw with an appropriate sampling instrument a representative portion of the material not less than 300 g. Divide it into three equal parts of about 100 g and transfer each to a clean and moisture-proof sample container which shall be closed air-tight ensuring that during this whole procedure the material and the sample are exposed to the atmosphere as little as possible. Each of these three samples shall be as individual sample representing that particular container.

C-3.2 There will be three sets of individual samples. Each set will contain a sample representing each selected container. One of these sets shall be marked for the purchaser, another for the supplier and the third shall be used as a referee set.

C-3.3 The referee set consisting of individual samples representing the selected containers shall bear the seal of both the purchaser and the supplier or their representatives. They shall be kept at a place and under conditions agreed to between the parties. The referee set shall be used in the case of dispute only.

C-4 NUMBER OF TESTS

C-4.1 Tests for the determination of caustic alkalinity, water-soluble aluminate and moisture content shall be conducted on each of the individual samples in a set.

C-4.2 The samples on which the individual tests are carried out, the average $\bar{X}$ and range (R) shall be calculated:

Where,

$$\bar{X} = \frac{\text{Sum of test results}}{\text{Number of tests}}$$

R = difference between maximum and minimum test results.

The lot shall be declared as conforming to the requirements of the individual sample if $\bar{X}$ — 0·6R is greater than or equal to the minimum value specified and $\bar{X}$ + 0.6R is less than or equal to the maximum value specified in Table 1.

C-5 CRITERIA FOR CONFORMITY

C-5.1 For Individual Samples

The lot shall be declared as conforming to the requirements of caustic alkalinity, water-soluble aluminate and moisture content if each of the individual samples separately satisfies the relevant requirements.

C-5.2 For Composite Sample

For declaring the conformity of the lot to the requirements of the characteristics tested on the composite sample, the test result for each characteristic shall satisfy the corresponding requirements of this specification.

ANNEX D

(*Foreword*)

EXPERTS WHO MADE SIGNIFICANT CONTRIBUTION TO THE DEVELOPMENT OF THIS STANDARD

Organization	*Representative(s)*
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