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IS 259 (1969): Ammonium alum [CHD 1: Inorganic Chemicals]



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(Reaffirmed 1999)

Indian Standard
SPECIFICATION FOR AMMONIUM ALUM
(*First Revision*)

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110 002

Indian Standard

SPECIFICATION FOR AMMONIUM ALUM

(First Revision)

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Indian Standard
SPECIFICATION FOR AMMONIUM ALUM
(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 1 March 1969, after the draft finalized by the Inorganic Chemicals (Miscellaneous) Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard was originally issued in 1950. In order to keep abreast with the progress made by the industry in this field, the Sectional Committee responsible for its preparation decided to revise it upgrading the quality of the technical grade. Pure and analytical reagent grades which are now being manufactured in the country have also been included.

0.3 Taking into consideration the views of producers, consumers and technologists, the Sectional Committee felt that the standard should be related to the manufacturing and trade practices followed in the country in this field. A few indigenous samples were analyzed before laying down this revised specification.

0.4 This standard contains clause **4.1** which calls for agreement between the purchaser and the supplier.

0.5 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 ammonium alum (aluminium ammonium sulphate), commercially known as ammonia alum.

*Rules for rounding off numerical values (revised).

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2. GRADES

2.1 There shall be three grades of the material, namely:

- a) *Technical (TECH)* — suitable for use in textile and paper industries;
- b) *Pure* — suitable for use in fine chemicals, photographic and pharmaceutical industries, in purification of water and in food preparations; and
- c) *Analytical Reagent (AR)* — suitable for use as a reagent in chemical analysis.

3. REQUIREMENTS

3.1 **Description** — The material shall be in the form of large colourless crystals, white granules or powder. It shall be free from visible impurities and other foreign matter.

3.2 The material shall also 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 6 of the table.

4. PACKING AND MARKING

4.1 **Packing** — The material shall be packed as agreed to between the purchaser and the supplier.

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

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

4.2.1 In case of analytical reagent grade, complete details of analysis in respect of the characteristics as laid down in Table 1 shall also appear on the label.

4.2.2 The product may also be marked with Standard mark.

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

TABLE 1 REQUIREMENTS FOR AMMONIUM ALUM

(Clause 3.2)

Sl No.	CHARACTERISTIC	REQUIREMENT FOR GRADE			METHOD OF TEST (REFERENCE TO CL No. IN APPENDIX A)
		TECH	PURE	AR	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Ammonium alum [$\text{Al}_2(\text{SO}_4)_3(\text{NH}_4)_2\text{SO}_4 \cdot 24\text{H}_2\text{O}$] content, per- cent by weight, <i>Min</i>	99.0	99.0	99.5	A-2
ii)	Matter insoluble in water, percent by weight, <i>Max</i>	0.15	0.02	0.01	A-3
iii)	Alkali and alkaline earths, percent by weight, <i>Max</i>	0.50	0.50	0.15	A-4
iv)	Arsenic (as As_2O_3), parts per million, <i>Max</i>	—	5	3	A-5
v)	Chloride (as Cl), per- cent by weight, <i>Max</i>	—	—	0.004	A-6
vi)	Fluoride (as F), percent by weight, <i>Max</i>	—	0.003	—	A-7
vii)	Heavy metals (as Pb), percent by weight, <i>Max</i>	—	0.002	0.001	A-8
viii)	Iron (as Fe), percent by weight, <i>Max</i>	0.006	0.002	0.001	A-9
ix)	Selenium (as Se), per- cent by weight, <i>Max</i>	—	0.003	—	A-10
x)	pH, not less than	2.9	2.9	3.0	A-11

5. SAMPLING

5.1 The method of drawing representative samples of the material, the number of tests to be performed and the criteria for conformity of the material to the requirements of this specification shall be as prescribed in Appendix B.

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APPENDIX A

(Clause 3.2)

ANALYSIS OF AMMONIUM ALUM

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (see IS : 1070-1960*) shall be employed in tests.

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

A-2. DETERMINATION OF AMMONIUM ALUM CONTENT [$\text{Al}_2(\text{SO}_4)_3(\text{NH}_4)_2\text{SO}_4 \cdot 24\text{H}_2\text{O}$]

A-2.0 General — Two methods are prescribed, namely:

- a) EDTA method, and
- b) gravimetric method.

Gravimetric method shall be the referee method in case of dispute.

A-2.1 EDTA Method

A-2.1.0 Outline of the Method — Ammonium aluminium sulphate is determined by reacting it with a known quantity of disodium ethylenediaminetetra acetate which is determined by back titration with zinc sulphate solution using dithizone as an indicator.

A-2.1.1 Reagents

A-2.1.1.1 Standard disodium ethylenediaminetetra acetate solution (EDTA) — 0.05 M. Dissolve 18.6 g of disodium ethylenediaminetetra acetate in sufficient water and make the volume to 1 000 ml in a volumetric flask.

A-2.1.1.2 Buffer solution (pH 4.5) — Dissolve 77.1 g of ammonium acetate and 57 ml of glacial acetic acid and make up with water to 1 000 ml.

A-2.1.1.3 Absolute alcohol — See IS : 321-1964†.

A-2.1.1.4 Dithizone indicator solution — Dissolve 25.6 mg of dithizone in 100 ml of absolute alcohol. It shall be prepared afresh.

*Specification for water, distilled quality (revised).

†Specification for absolute alcohol (revised).

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A-2.1.1.5 Standard zinc sulphate solution — (0.05 M) — Dissolve 14.37 g of zinc sulphate ($\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$), in water and make up the solution to exactly 1 000 ml.

A-2.1.2 Procedure — Weigh accurately about 1 g of the material and dissolve in 50 ml of water. Add, in the order given and with mixing after each addition, 50 ml of disodium ethylenediaminetetra acetate solution, 20 ml of buffer solution, 50 ml of absolute alcohol and 2 ml of dithizone indicator solution. Titrate the excess of EDTA solution with standard zinc sulphate solution to a bright rose-pink colour. Carry out a blank determination also simultaneously. Each ml of 0.05 M disodium ethylenediaminetetra acetate is equivalent to 22.67 mg of aluminium ammonium sulphate [$\text{Al}_2(\text{SO}_4)_3(\text{NH}_4)_2\text{SO}_4 \cdot 24\text{H}_2\text{O}$].

A-2.1.3 Calculation

Ammonium Alum [$\text{Al}_2(\text{SO}_4)_3(\text{NH}_4)_2\text{SO}_4 \cdot 24\text{H}_2\text{O}$],

$$\text{percent by weight} = \frac{45.34 V M}{W}$$

where

V = volume in ml of disodium ethylenediaminetetra acetate solution consumed,

M = molarity of disodium ethylenediaminetetra acetate solution, and

W = weight in g of the material taken for the test.

A-2.2 Gravimetric Method

A-2.2.0 Outline of the Method — Aluminium is precipitated by ammonium hydroxide, ignited and calculated as aluminium ammonium sulphate.

A-2.2.1 Reagents

A-2.2.1.1 Ammonium chloride — solid.

A-2.2.1.2 Dilute sulphuric acid — approximately 1 N.

A-2.2.1.3 Methyl red indicator — prepared by dissolving 0.15 g of methyl red in 500 ml of water.

A-2.2.1.4 Dilute ammonium hydroxide — approximately 10 percent (w/w).

A-2.2.1.5 Ammonium nitrate solution — approximately 2 percent (w/v).

A-2.2.2 Procedure — Weigh accurately about 0.5 g of the material and dissolve in 100 ml of water. Filter to remove any undissolved matter

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and thoroughly wash the filter paper with hot water. Add to the filtrate and washings about 5 g of ammonium chloride and 10 ml of dilute sulphuric acid. Heat to boiling and add a few drops of methyl red indicator. Add ammonium hydroxide to the boiling liquid drop by drop till the colour changes to distinct yellow. Continue boiling for two minutes and filter immediately through a Whatman filter paper No. 41. Reserve the filtrate for test under A-4. Wash the precipitate with hot ammonium nitrate solution till free from chlorides. Dry the residue carefully and ignite in a tared silica crucible to a constant weight at about 1100° to 1200°C.

A-2.2.3 Calculation

$$\text{Aluminium ammonium sulphate } [\text{Al}_2 (\text{SO}_4)_3 (\text{NH}_4)_2 \text{SO}_4 \cdot 24\text{H}_2\text{O}]$$

$$\text{percent by weight} = \frac{889.4 W_1}{W}$$

where

W_1 = weight in g of the residue; and

W = weight in g of the material taken for the test.

A-3. DETERMINATION OF MATTER INSOLUBLE IN WATER

A-3.1 Procedure — Accurately weigh about 10 g of the material. Dissolve in 100 ml of water by warming, if necessary, and by stirring the solution well. Filter through a tared filter paper or through a tared sintered glass crucible G 4 or Gooch crucible, wash thoroughly the residue till it is free from all soluble compounds and dry at 105° to 110°C to constant weight.

A-3.2 Calculation

$$\text{Matter insoluble in water, percent by weight} = 100 \frac{W_1}{W}$$

where

W_1 = weight in g of the residue, and

W = weight in g of the material taken for the test.

A-4. DETERMINATION OF ALKALI AND ALKALINE EARTHS

A-4.1 Procedure — Evaporate the filtrate obtained in A-2.2.2 in a tared platinum evaporating dish to dryness. Ignite and weigh till constant weight. Express it as percentage of the material taken for the test.

A-5. DETERMINATION OF ARSENIC

A-5.1 Dissolve 2 g of the material in 50 ml of water. Take 25 ml of this solution containing 1 g of the material for the test and carry out the test for arsenic as prescribed in IS : 2088-1962* using for comparison a stain obtained with 0.005 mg of arsenic trioxide (as As_2O_3) in case of pure grade and 0.003 mg in case of analytical reagent grade.

A-5.2 The limit prescribed in Table I shall be taken as not having been exceeded if the length of the stain as well as the intensity of its colour produced in the test with the material is not greater than those produced in the control tests.

A-6. DETERMINATION OF CHLORIDE

A-6.0 Outline of the Method — Chloride is determined by turbidimetric comparison with solution containing a known quantity of chloride.

A-6.1 Apparatus

A-6.1.1 Nessler Cylinders — 50 ml capacity.

A-6.2 Reagents

A-6.2.1 Dilute Nitric Acid — approximately 5 N.

A-6.2.2 Silver Nitrate Solution — approximately 5 percent (w/v).

A-6.2.3 Standard Chloride Solution — Dissolve 165 mg of sodium chloride in water and dilute to exactly 100 ml. Transfer 10 ml of this solution to a 100-ml volumetric flask and dilute to the mark with water and mix. One millilitre of this solution is equivalent to 0.1 mg of chloride (as Cl).

A-6.3 Procedure — Weigh accurately 2.5 g of the material and dissolve in 50 ml of water. Add 1 ml of dilute nitric acid and 1 ml of silver nitrate solution. Stir immediately with a glass rod and set aside for five minutes. Carry out a control test with one millilitre of standard chloride solution in place of the sample and the same quantities of other reagents in the same total volume of the reaction mixture. Compare the turbidity in the two sets.

A-6.3.1 The limits prescribed in Table I shall be taken as having not been exceeded if the opalescence produced in the sample solution is not greater than that produced in the control test.

*Modified Gutzeit method of determination of arsenic. (Since revised).

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A-7. DETERMINATION OF FLUORIDE

A-7.0 Outline of the Method — Fluoride is separated from the interfering elements as hydrofluosilicic acid by distillation with perchloric acid and is titrated with standard thorium nitrate solution using sodium alizarin sulphonate as indicator. At the end point a pink colour of a thorium lake appears.

A-7.1 Apparatus

A-7.1.1 Nessler Cylinders — 50 ml capacity.

A-7.1.2 Distilling Apparatus — A distilling flask connected with a condenser and carrying a thermometer, a capillary tube and a dropping funnel.

A-7.2 Reagents

A-7.2.1 Lime Suspension — Carefully shake about 56 g of low-fluorine calcium oxide (about 2 parts per million F) with 250 ml of water, and add 250 ml of 60 percent perchloric acid slowly and with stirring. Add a few glass beads and boil to copious fumes of perchloric acid. Cool, add 200 ml of water and boil again. Repeat the dilution and boiling once more. Cool and dilute considerably and filter through a sintered glass crucible, if precipitate of silica appears. Pour the clear solution with stirring into 1 000 ml of sodium hydroxide solution (10 percent). Allow the precipitate to settle and siphon off the supernatant liquid. Remove the sodium salts from the precipitate either by washing 5 times in large centrifuge bottles, shaking the mass thoroughly each time or by filtering and washing the precipitate. Finally, shake the precipitate into a suspension and dilute to 2 000 ml. Store in paraffin-lined bottles and shake well before use.

NOTE — 100 ml of this suspension should give no appreciable fluoride blank when evaporated, distilled and titrated as in A-7.3.

A-7.2.2 Perchloric Acid — 60 percent (w/v).

A-7.2.3 Sodium Hydroxide Solution — 2.5 N and 0.05 N.

A-7.2.4 Dilute Sulphuric Acid — 50 percent (v/v).

A-7.2.5 Hydrogen Peroxide — 30 percent (w/w).

A-7.2.6 Silver Perchlorate Solution — 50 percent (w/v).

A-7.2.7 Dilute Hydrochloric Acid — approximately 0.1 N.

A-7.2.8 Thorium Nitrate Solution — Dissolve 250 mg of thorium nitrate [$\text{Th}(\text{NO}_3)_4 \cdot \text{H}_2\text{O}$] in 1 000 ml of water.

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A-7.2.9 Sodium Fluoride Solution — Dry about 500 mg of sodium fluoride at 200°C for 4 hours. Dissolve 222 mg of the dried material, accurately weighed, in sufficient water to make 100 ml. Dilute 10 ml of this solution to 1 000 ml with water. Each millilitre of this solution contains 0.01 mg of fluorine (as F).

A-7.2.10 Sodium Alizarin Sulphonate Solution — Dissolve 100 mg in 100 ml of water and filter.

A-7.3 Procedure — Add to the distilling flask 1.67 g of the sample, accurately weighed, and 25 ml of dilute sulphuric acid. Distil until the temperature reaches 160°C, then maintain the temperature between 160° and 165°C by adding water from the funnel and collect about 300 ml of the distillate. Oxidize the distillate by continuously adding 2 to 3 ml of fluorine-free hydrogen peroxide (to remove sulphites). Allow to stand for a few minutes, and evaporate in a platinum dish with an excess of lime suspension. Ignite at 600°C, then cool and wet the residue with about 10 ml of water. Cover the dish with a watch-glass and cautiously introduce under cover just sufficient perchloric acid to dissolve the residue. Add contents of the dish, through the dropping funnel of the distilling apparatus (the distilling flask should contain a few glass beads), using a total of 20 ml of perchloric acid for dissolving the residue and transferring the solution. Add 10 ml of water and a few drops of silver perchlorate solution through the dropping funnel. Continue the distillation until 70 ml has been collected, dilute the distillate to 80 ml and mix well. Place 40 ml of the solution in a Nessler cylinder. In another similar Nessler cylinder place 40 ml of water as a control. Add to each cylinder 0.1 ml of sodium alizarin sulphonate solution and mix well. Add dropwise and with stirring 0.05 N sodium hydroxide solution to the cylinder containing the distillate until its colour just matches that of the control which is faintly pink. Then add to each cylinder exactly 1 ml of dilute hydrochloric acid, and mix well. From a burette graduated in 0.05 ml, add to the cylinder containing the distillate enough thorium nitrate solution so that, after mixing, the colour of the liquid just changes to a faint pink. Note the volume of the solution added; add exactly the same volume to the control and mix well. Now add to the control standard sodium fluoride solution from a burette to make the colour of the two cylinders match after dilution to the same volume. Mix well and allow the air bubbles to escape before making the final colour comparison. Check the end point by adding 1 or 2 drops of standard sodium fluoride solution to the control test. A distinct change in colour should take place.

A-7.3.1 The limit as prescribed in Table 1 shall be taken as having not exceeded if not more than 2.5 ml of the standard sodium fluoride solution is required.

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A-8. DETERMINATION OF HEAVY METALS

A-8.0 Outline of the Method — Heavy metals are determined by the dark coloration produced with sodium sulphide solution.

A-8.1 Apparatus

A-8.1.1 Nessler Cylinders — 50 ml capacity.

A-8.2 Reagents

A-8.2.1 Standard Lead Solution — Dissolve 160 mg of lead nitrate in 5 ml of concentrated nitric acid and dilute with water to make the solution to 100 ml. Dilute 1 ml of this solution to 100 ml with water immediately before use. One millilitre of this solution is equivalent to 0.01 mg of lead (as Pb).

A-8.2.2 Sodium Sulphide Solution — 5 percent (w/v).

A-8.3 Procedure — Dissolve 4 g of the material in water to produce 40 ml of the solution. Transfer 30 ml of this solution to Nessler cylinder and dilute to 50 ml. Transfer the remaining 10 ml of the solution to another Nessler cylinder and 4 ml and 2 ml of standard lead solution for pure grade and analytical reagent grade respectively, and dilute to 50 ml. To both the solutions, add 2 drops of sodium sulphide solution and compare the colour produced in the two cylinders.

A-8.3.1 The limit prescribed in Table 1 shall be taken as having not been exceeded if the colour produced in the test is not deeper than that produced in the control test.

A-9. DETERMINATION OF IRON

A-9.0 Outline of the Method — Iron is determined colorimetrically by visual comparison using potassium thiocyanate.

A-9.1 Apparatus

A-9.1.1 Nessler Cylinders — 50 ml capacity.

A-9.2 Reagents

A-9.2.1 Dilute Hydrochloric Acid — Approximately 5 N.

A-9.2.2 Butanolic Potassium Thiocyanate Solution — Dissolve 10 g of potassium thiocyanate in 10 ml of water. Add sufficient *n*-butanol to make up to 100 ml and shake vigorously until the solution is clear.

A-9.2.3 Ammonium Persulphate

A-9.2.4 Standard Iron Solution — Dissolve 0.702 g of ammonium ferrous sulphate in a few millilitres of water, add 10 ml of dilute sulphuric acid (10 percent v/v). Dilute the solution to one litre. Take 10 ml of this solution and dilute it exactly to 100 ml. One millilitre of this solution is equivalent to 0.01 mg of iron (as Fe). The diluted solution shall be freshly prepared.

A-9.3 Procedure — Dissolve 1.0 g of the material in 10 ml of water. Add one millilitre of dilute hydrochloric acid, 30 mg of ammonium persulphate and 15 ml of butanolic potassium thiocyanate solution. Shake vigorously for 30 seconds and allow to separate. Carry out a control test proceeding exactly as for the material under test, using the following quantities of standard iron solution and compare the intensity of the colour produced in the butanolic layers of the two cylinders:

- a) 6 ml for technical grade;
- b) 2 ml for pure grade; and
- c) 1 ml for analytical reagent grade.

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

A-10. DETERMINATION OF SELENIUM

A-10.0 Outline of the Method — Selenium is determined colorimetrically by using ascorbic acid with which it gives a pink colour.

A-10.1 Apparatus

A-10.1.1 Nessler Cylinders — 50 ml capacity.

A-10.2 Reagents

A-10.2.1 Dilute Hydrochloric Acid — 5 N.

A-10.2.2 Dilute Nitric Acid — 5 N.

A-10.2.3 Standard Selenium Solution — Dissolve 120 mg of metallic selenium in 100 ml of dilute nitric acid by gently warming on a water-bath and make up the volume to 1 000 ml. Transfer 12.5 ml of this solution to a 500-ml volumetric flask and make up the volume. One millilitre of this solution is equivalent to 0.003 mg of selenium (as Se).

A-10.2.4 Ascorbic Acid — solid.

A-10.3 Procedure — Dissolve 2 g of the material in 40 ml of dilute hydrochloric acid and transfer into a Nessler cylinder. In another Nessler cylinder transfer 20 ml of standard selenium solution and add

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20 ml of hydrochloric acid and mix. Place the two Nessler cylinders containing the sample solution and standard solution at 40°C in a water-bath, and heat until the temperature of the solution reaches 40°C. To each cylinder add 400 mg of ascorbic acid, stir until dissolved and maintain the temperature at 40°C for another 30 minutes. Cool the solutions, dilute with water to 50 ml and mix.

A-10.3.1 The limit prescribed in Table 1 shall be taken as having not been exceeded if any colour produced in the sample solution is not greater than that produced with standard solution.

A-11. DETERMINATION OF pH**A-11.1 Apparatus**

A-11.1.1 A suitable pH-meter complete with all accessories.

A-11.2 Procedure -- Weigh 5 g of the material and dissolve in 20 ml of carbon dioxide-free water and dilute to 100 ml. Determine the pH of the solution with the help of pH-meter, using glass electrodes.

A P P E N D I X B*(Clause 5.1)***SAMPLING OF AMMONIUM ALUM****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 The sampling implement shall be clean and dry when used.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling implement and the containers for samples from adventitious contaminations.

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed thoroughly by suitable means.

B-1.5 The samples shall be placed in suitable, clean, dry and air-tight glass containers which shall be sealed air-tight after filling and marked with full details of sampling.

B-2. SCALE OF SAMPLING

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

B-2.1.1 For ascertaining the conformity of the material in a lot to the requirements of the specification, tests shall be carried out for each lot separately. The number of containers to be selected at random from lots of different sizes shall be in accordance with Table 2.

TABLE 2 SCALE OF SAMPLING

Lot Size (<i>N</i>)	SAMPLE SIZE (<i>n</i>)
(1)	(2)
Up to 25	3
26 „ 50	4
51 „ 100	5
101 and above	7

B-2.1.2 Random number tables shall be used for selecting the containers. In case such tables are not available, the following procedure is recommended for use:

Starting from any container at random, count them in one order as 1, 2, 3, ..., etc, up to r and so on where r is the integral part of N/n . (N and n being the lot size and sample size respectively). Every r th container thus counted shall be withdrawn to constitute the sample.

B-3. PREPARATION OF TEST SAMPLES

B-3.1 From each of the containers selected, draw a small representative portion of ammonium alum approximately 50 g in weight.

B-3.2 Out of these portions, a small but equal quantity of the material shall be taken and mixed thoroughly by suitable means to form a composite sample of weight about 100 g. The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

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B-3.3 From the remaining portion of ammonium alum from each container, about 30 g of the material shall be drawn and be divided into three equal parts which shall then be transferred to three separate bottles with full identification particulars of the sample. The material in each bottle constitute individual sample. One of these three sets (each set containing n bottles representing n containers sampled) shall be for the purchaser, another for the supplier and the third for the referee.

B-3.4 The referee samples consisting of a composite sample and a set of individual samples shall bear the seal of both the purchaser and the supplier. They 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 ammonium alum content shall be performed on each of the individual samples.

B-4.2 Tests for the determination of all the remaining characteristics given in Table 1 shall be carried out on the composite sample.

B-5. CRITERION FOR CONFORMITY

B-5.1 For Ammonium Alum Content — From the set of individual test results for ammonium alum content the average \bar{X} and the range R of the test results shall be computed. (Range R is defined as the difference between the maximum and the minimum test results.)

B-5.1.1 A lot shall be declared as conforming to the requirements of ammonium alum content if $(\bar{X} - 0.6R)$ is greater than or equal to 99.0 in the case of technical grade and pure grade and 99.5 in the case of analytical reagent grade.

B-5.2 For declaring the conformity of the lot to the requirements of all the other characteristics tested on the composite sample, the test results shall meet the corresponding requirements specified in Table 1.

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Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002

Telephones: 323 0131, 323 3375, 323 9402

Fax : 91 11 3234062, 91 11 3239399, 91 11 3239382

Telegrams : Manaksanstha
(Common to all Offices)

Central Laboratory:

Plot No. 20/9, Site IV, Sahibabad Industrial Area, SAHIBABAD 201010

Telephone

8-77 00 32

Regional Offices:

Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002 323 76 17

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

Northern : SCO 335-336, Sector 34-A, CHANDIGARH 160022 60 38 43

Southern : C.I.T. Campus, IV Cross Road, CHENNAI 600113 235 23 15

†Western : Manakalaya, E9 Behind Marol Telephone Exchange, Andheri (East),
MUMBAI 400093 832 92 95

Branch Offices:

'Pushpak', Nurmohamed Shaikh Marg, Khanpur, AHMEDABAD 380001 550 13 48

‡Peenya Industrial Area, 1st Stage, Bangalore - Tumkur Road,
BANGALORE 560058 839 49 55

Gangotri Complex, 5th Floor, Bhadbhada Road, T. T. Nagar, BHOPAL 462003 55 40 21

Plot No. 62-63, Unit VI, Ganga Nagar, BHUBANESHWAR 751001 40 36 27

Kalaikathir Buildings, 670 Avinashi Road, COIMBATORE 641037 21 01 41

Plot No. 43, Sector 16 A, Mathura Road, FARIDABAD 121001 8-28 88 01

Savitri Complex, 116 G. T. Road, GHAZIABAD 201001 8-71 19 96

53/5 Ward No. 29, R. G. Barua Road, 5th By-lane, GUWAHATI 781003 54 11 37

5-8-58C, L. N. Gupta Marg, Nampally Station Road, HYDERABAD 500001 20 10 83

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

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

Seth Bhawan, 2nd Floor, Behind Leela Cinema, Naval Kishore Road,
LUCKNOW 226001 23 89 23

Patliputra Industrial Estate, PATNA 800013 26 23 05

T. C. No. 14/1421, University P. O. Palayam,
THIRUVANANTHAPURAM 695034 6 21 17

NIT Building, Second Floor, Gokulpat Market, NAGPUR 440010 52 51 71

Institution of Engineers (India) Building, 1332 Shivaji Nagar, PUNE 411005 32 36 35

*Sales Office is at 5 Chowringhee Approach, P. O. Princep Street,
CALCUTTA 700072 27 10 85

†Sales Office is at Novelty Chambers, Grant Road, MUMBAI 400007 309 65 28

‡Sales Office is at 'F' Block, Unity Building, Narashimaraja Square,
BANGALORE 560002 222 39 71