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मानक

IS 1113 (1965): Ammonium chloride, technical and pure [CHD 1: Inorganic Chemicals]



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Indian Standard

SPECIFICATION FOR AMMONIUM CHLORIDE, TECHNICAL AND PURE

(Revised)

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

July 1965

Indian Standard

SPECIFICATION FOR AMMONIUM CHLORIDE, TECHNICAL AND PURE

(Revised)

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Indian Standard SPECIFICATION FOR AMMONIUM CHLORIDE, TECHNICAL AND PURE (Revised)

0. FOREWORD

0.1 This Indian Standard (Revised) was adopted by the Indian Standards Institution on 31 January 1965, after the draft finalized by th Acids and Fertilizers Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard was first published in 1957. Comments were received that the requirement for 'heavy metals including iron' as given in the standard was vague and the method of test for the same was also not compatible with the requirement prescribed. The Committee responsible for the preparation of this standard decided to revise the specification. In this revision, separate requirements for cosmetic grade have been included. The requirements for heavy metal including iron except for cosmetic grade have been replaced by a limit for iron only. Also, the methods of sampling and test have been reviewed and modified suitably.

0.3 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 test for ammonium chloride, technical and pure, intended for use in soda ash industry, fermentation, soldering, galvanizing, tinning trades, dry cells and cosmetics.

1.1.1 This standard does not apply to the material intended for use as fertilizer for which a separate standard IS: 1114-1964[†] has been drawn up.

^{*}Rules for rounding off numerical values (revised).

[†]Specification for ammonium chloride, fertilizer grade (revised).

2. GRADES AND TYPES

2.1 Grades - The material shall be of two grades, namely:

- a) Technical For use in soda ash industry, fermentation, soldering, galvanizing, and tinning trades; and
- b) Pure
- 2.2 Types The pure grade shall be of two types, namely:
 - a) Type 1 For use in dry cells, and
 - b) Type 2 For use in cosmetic industry.

3. REQUIREMENTS

3.1 Description — Ammonium chloride, technical and pure, shall be in the form of white crystals, pellets or bars friable and free from hard caking, perceptible odour and also any visible impurities.

3.2 The material shall also comply with the requirements specified in Table 1 when tested according to the methods given in Appendix A. Reference to the relevant clauses of Appendix A is given in col 6 of the table.

3.3 Special Requirement for Dry Cell Manufacture—The material intended for use in the manufacture of dry cells shall be free from copper, cobalt, nickel, arsenic and antimony, when tested by the methods prescribed in Appendix B.

4. PACKING AND MARKING

4.1 The material shall be packed in multi-walled paper or jute b_gs lined with polyethylene films (see IS: $2508 - 1963^*$) or in such other containers as agreed to between the purchaser and the supplier.

4.2 The containers shall be securely closed and marked with the following information:

- a) Name of the material;
- b) Name of the manufacturer;
- c) Weight of the material in the container;
- d) Recognized trade-mark, if any; and
- e) Month and year of manufacture.

^{*}Specification for low density polyethylene films.

31 No. CHARAOTEBISTIC	REQUIREMENTS FOR			METHODS OF	
	Technical Grade	Pure Grade		TEST (REF TO CL NO. IN APPENDIX)	
			Type 1	Type 2	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Moisture, persent by weight, Max	1.0	0.2	0.2	A-3
ii)	Ammonium chloride (as NH ₄ Cl), percent by weight (on dry basis), <i>Min</i>	98•5	9 9 •5	99-5	A-4
iii)	Iron (as Fe), percent by weight, Max	0.01	0.01		A-5
iv)	Sulphates [as (NH ₄) ₂ SO ₄], percent by weight, Max	0.3	0.10	0-10	A-6
V)	Total sulphated residue on sublimation, per- cent by weight, Max	_	0.3	0.3	A-7
vi)	pH		4.5 to 5.2	4.5 to 5.0	A-8
vii)	Matter insoluble in water, percent by weight, Max	0.5	0.1	0 .1	A-9
viii)	Heavy metals including iron (as Pb), parts per million, Max		_	10	A-10
i x)	Arsenic (as As_3O_3), parts per million, Max			10	A-11

TABLE 1 REQUIREMENTS FOR AMMONIUM CHLORIDE, TECHNICAL AND PURE

(Clause 3.2)

4.2.1 The containers may also be marked with the Standard Mark

NOTE — 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 Standard 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 BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5. SAMPLING

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

APPENDIX A

(*Clause* 3.2)

ANALYSIS OF AMMONIUM CHLORIDE, TECHNICAL AND PURE

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. PREPARED SAMPLE

A-2.1 Mix the sample well by shaking the bottle several times and transfer a portion immediately to a wide-mouth bottle and stopper it. Take care that no pieces of cork or sealing wax get mixed with the material. Do not expose the *prepared sample* to an atmosphere containing acid or alkaline fumes.

A-3. DETERMINATION OF MOISTURE

A-3.1 Procedure — Weigh accurately about 5 g of the *prepared sample* in a weighed shallow porcelain dish and dry for 24 hours in a vacuum desiccator over sulphuric acid and reweigh. Preserve the dried material for subsequent test.

A-3.2 Calculation

Moisture, percent by weight =
$$100 \frac{W_1}{W_2}$$

where

 $W_1 = \text{loss in weight in g on drying, and}$ $W_2 = \text{weight in g of the prepared sample taken for the test.}$

^{*}Specification of water, distilled quality (revised).

A-4. DETERMINATION OF AMMONIUM CHLORIDE

A-4.1 Apparatus — The apparatus as assembled is shown in Fig. 1. It consists of a flask A of 1 000 ml capacity fitted with a rubber stopper through which passes one end of the connecting bulb tube B. The other end of the bulb tube B is connected to the condenser C by a rubber stopper and the lower end of the condenser C is attached by means of a rubber tubing to a dip tube D which dips into a beaker E of 250 ml capacity.



FIG. 1 APPABATUS FOR DETERMINATION OF AMMONIUM CHLORIDE

A-4.2 Reagents

A-4.2.1 Standard Sulphuric Acid – 0.1 N.

A-4.2.2 Methyl Red Indicator Solution — Dissolve 0.15 g of methyl red in 500 ml of water.

A-4.2.3 Magnesium Oxide - freshly ignited and free from carbonate.

A-4.2.4 — Standard Sodium Hydroxide Solution 0.1 N.

A-4.3 Procedure — Weigh accurately about 0.2 g of the dried material (A-3,1) and transfer to a beaker. Dissolve in about 20 ml of water and transfer the solution to the distillation flask A and add about 500 ml of water. Assemble the apparatus as shown in Fig. 1, with the tip of the dip tube D dipping in dilute sulphuric acid contained in the beaker E to which a few drops of methyl red indicator have been added. Add about 10 g of the freshly ignited magnesium oxide and distil at least one third of the total volume of the liquid in flask A to ensure complete distillation. Lower the beaker E until the end of the dip tube D is out of the dilute sulphuric acid. Shut off the burner and when the flask A becomes cool, detach it from the condenser and rinse the condenser thoroughly with water into the beaker E. Add two or three drops of methyl red indicator and titrate with standard sodium hydroxide solution.

A-4.3.1 Carry out a blank test using all the reagents in the same quantities but without the material.

A-4.4 Calculation

Ammonium chloride, percent by
weight (on dry basis) =
$$\frac{5 \cdot 350 (B-A) N}{W}$$

where

- B = volume in ml of standard sodium hydroxide solution used in the blank determination,
- A = volume in ml of standard sodium hydroxide solution used in the test with the material,
- N = normality of standard sodium hydroxide solution, and
- W = weight in g of the dried material taken for the test.

A-5. TEST FOR IRON

A-5.1 Apparatus

A-5.1.1 Nessler Tubes - 50 ml capacity.

A-5.2 Reagents

A-5.2.1 Dilute Hydrochloric Acid - approximately 4 N.

A-5.2.2 Ammonium persulphate

A-5.2.3 Butanolic Potassium Thiocyanate Solution — Dissolve 10 g of potassium thiocyanate in 10 ml of water. Add sufficient *n*-butanol to make the volume 100 ml and shake vigorously until the solution is clear.

A-5.2.4 Standard Iron Solution — Dissolve 7.02 g of ferrous ammonium sulphate $[FeSO_4. (NH_4)_2SO_4, 6H_2O]$ in water in a 1000-ml flask, add 10 ml of concentrated sulphuric acid conforming to IS : 266 - 1961* and dilute with water to the mark. When required for use, dilute 50 ml of the solution to 500 ml in a volumetric flask. One millilitre of the diluted solution contains 0.1 mg of iron (as Fe).

A-5.3 Procedure — Weigh 1.000 g of the material and transfer into a Nessler tube. Add 25 ml of water, 5 ml of dilute hydrochloric acid, 30 mg of ammonium persulphate and 15 ml of butanolic potassium thiocyanate solution. Make up to 50 ml with water, shake vigorously and allow the two layers to separate. Carry out a control test in another Nessler tube using 1 ml of standard iron solution in place of the material and the same quantities of other reagents.

A-5.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of the colour produced with the material is not greater than that produced in the control test.

A-6. TEST FOR SULPHATES

A-6.1 Apparatus

A-6.1.1 Nessler Tubes — 50 ml capacity.

A-6.2 Reagents

A-6.2.1 Dilute Hydrochloric Acid - approximately 5 N.

A-6.2.2 Barium Chloride Solution - approximately 10 percent.

A-6.2.3 Standard Sulphate Solution — Dissolve 1 000 g of ammonium sulphate in water and make up the volume to 1 000 ml. Pipette 10 ml of this solution and again dilute to 100 ml. One millilitre of this solution contains 0.1 mg of sulphate [as $(NH_4)_2SO_4$].

A-6.3 Procedure — Dissolve 1.000 g of the material in sufficient quantity of water and make up the volume to 100 ml. Transfer 30 ml (for technical grade or 10 ml (for pure grade) of this solution to a Nessler tube, and add

^{*}Specification for sulphuric acid (revised).

1 ml of dilute hydrochloric acid. Carry out a control test in another Neuler tube using 3 ml of standard sulphate solution and 1 ml of dilute hydrochloric acid. Add 1 ml of barium chloride solution to both the tubes, make up the volume to 50 ml and compare the turbidity produced after 5 minutes.

A-6.3.1 The limit prescribed in Table 1 shall be taken to be as not having been exceeded if the turbidity produced in the test with the material is not greater than that produced in the control test.

A-7. DETERMINATION OF TOTAL SULPHATED RESIDUE ON SUBLIMATION

A-7.1 Reagent

A-7.1.1 Concentrated Sulphuric Acid -- conforming to IS: 266 - 1961*.

A-7.2 Procedure—Weigh accurately about 5 g of the dried material (A-3.1) into a weighed crucible, heat it till most of the material sublimes off. Add about one ml of concentrated sulphuric acid. Mix with the residue and finally ignite to constant weight.

A-7.3 Calculation

Total sulphated residue after sublimation, percent by weight = $100 \frac{W_1}{W_2}$

where

 W_1 = weight in g of the residue, and

 W_2 = weight in g of the dried material taken for the test.

A-8. DETERMINATION OF *pH*

A-8.1 Procedure — Weigh about 5 g of the material and transfer to a 250-ml beaker of resistant glass. Add about 100 ml of carbon dioxide-free water, stir well and set aside for a short while. Determine the pH of the solution by any standard method using a potentiometric instrument.

A-8.1.1 In case of dispute, the value given by a glass electrode shall be taken as correct.

A-9. DETERMINATION OF MATTER INSOLUBLE IN WATER

A-9.1 Procedure — Weigh accurately about 20 g of the material, dissolve in 150 ml of water and heat on a water-bath for about an hour. Take a

^{*}Specification for sulphuric acid (revised).

filter paper wash it well with water, dry to constant weight at 105°C. Filter any insoluble matter, wash it with water and dry at $105^{\circ} \pm 2^{\circ}$ C to constant weight.

A-9.2 Calculation

Matter insoluble in water, percent by weight = $100 \frac{W_1}{W_2}$

where

 W_1 = weight in g of the residue, and

 W_2 = weight in g of the material taken for the test.

A-10. DETERMINATION OF HEAVY METALS INCLUDING IRON

A-10.1 Apparatus

A-10.1.1 Nessler Tubes - 50 ml capacity.

A-10.2 Reagents

A-10.2.1 Concentrated Hydrochloric Acid — conforming to IS : 265-1962*

A-10.2.2 Ammonium Chloride

A-10.2.3 Acetic Acid - 1 N.

A-10.2.4 Standard Lead Solution — Dissolve 1.60 g of lead nitrate in water, add one. millilitre of concentrated nitric acid (conforming to IS: 264-1950† and make up the volume to 1 000 ml. Pipette out 10 ml of this solution and dilute again to 1 000 ml with water. One millilitre of this solution contains 0.01 mg of lead (as Pb).

A-10.2.5 Hydrogen Sulphide Solution — freshly prepared, saturated solution.

A-10.3 Procedure — Mix 2 g of the material with 10 ml of water and add just sufficient concentrated hydrochloric acid to dissolve the material. Evaporate to dryness on a steam-bath and dissolve the residue in about 30 ml of water. Transfer the solution to a Nessler tube, add 1 g of ammonium chloride and 1 ml of acetic acid. Carry out a control test in the other Nessler tube using 2 ml of standard lead solution in place of the material, add 1 g of ammonium chloride and 1 ml of acetic acid. To each Nessler tube add 10 ml of hydrogen sulphide solution, dilute to the

^{*}Specification for hydrochloric acid (revised).

[†]Specification for nitric acid.

mark and shake well, compare the intensity of colour produced in the two tubes.

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

A-11. DETERMINATION OF ARSENIC

A-11.1 Preparation of Solution — Accurately weigh 10 g of the material and dissolve in 30 ml of water. Transfer it to the Gutzeit bottle and proceed as described in IS : 2088-1962*, using for comparison a stain obtained with 0.01 mg of arsenic trioxide (as As_2O_3).

APPENDIX B

(Clause 3.3)

TEST FOR COPPER, COBALT, NICKEL, ARSENIC AND ANTIMONY

B-1. TEST FOR COPPER

B-1.1 Reagents

B-1.1.1 Ammonium Hydroxide — 20 percent (w/w).

B-1.1.2 Glacial Acetic Acid

B-1.1.3 Potassium Ferrocyanide Solution — Dissolve 50 g of potassium ferrocyanide in water and dilute to 1 litre.

B-1.2 Procedure — Dissolve 500 g of the material in 200 ml of water, add 5 ml of ammonium hydroxide and boil. Allow to settle for 30 minutes and filter while hot, using a medium texture filter paper and wash the precipitate a few times with hot water. To the filtrate add 3 ml of glacial acetic acid followed by 5 to 10 drops of potassium ferrocyanide solution.

B-1.2.1 The material shall be considered to be free from copper if no brownish red colouration develops after the addition of potassium ferro-cyanide solution.

B-2. TEST FOR COBALT AND NICKEL

B-2.1 Reagents

B-2.1.1 Concentrated Hydrochloric Acid — conforming to IS : 265-1962[†].

^{*}Modified Gutzeit method of test for arsenic.

[†]Specification for hydrochloric acid (*revised*).

B-2.1.2 Concentrated Sulphuric Acid -- conforming to IS : 266-1961*.

B-2.1.3 Hydrogen Sulphide Gas

B-2.1.4 Concentrated Nitric Acid - conforming to IS: 264-1950[†].

B-2.1.5 Ammonium Hydroxide — 20 percent (w/w).

B-2.1.6 Dilute Ammonium Hydroxide — approximately 2 N.

B-2.2 Procedure — Weigh 1000 g of the material in a porcelain casserole and heat over a low flame until 1 to 2 g of the material remain. Allow to cool, take up with 2 ml of concentrated hydrochloric acid and heat gently. Add 5 to 10 ml of water and 5 ml of concentrated sulphuric acid. Evaporate to fumes, allow to cool and take up with 25 ml of water and filter. Heat the filtrate to boiling and pass hydrogen sulphide gas through the hot solution and filter. Boil the filtrate to drive off hydrogen sulphide. Add 3 to 4 drops of concentrated nitric acid, neutralize with ammonium hydroxide, heat and filter. Wash the precipitate with warm dilute ammonium hydroxide and boil to drive off ammonia. Make the solution slightly acidic with concentrated hydrochloric acid and pass hydrogen sulphide gas. Make the solution just alkaline with ammonium hydroxide.

B-2.2.1 The material shall be considered to be free from cobalt and nickel if no black precipitate is formed on passing hydrogen sulphide in the alkaline solution, after the separation of iron.

B-3. TEST FOR ARSENIC

B-3.1 Procedure — Dissolve 10.0 g of the material in about 30 ml of water and carry out the test as prescribed in IS: 2088-1962[‡].

B-3.1.1 The material shall be considered to be free from arsenic if no stain is produced on the sensitized mercuric bromide paper.

B-4. TEST FOR ANTIMONY

B-4.1 Reagents

B-4.1.1 Dilute Ammonium Hydroxide — approximately 5 percent (w/v).

B-4.1.2 Gold Chloride ($AuCl_3$ HC1,4H₂O) Solution – approximately one percent (w/v).

B-4.2 Procedure—Carry out the test as prescribed in **B-3.1** but dip the sensitized mercuric paper in dilute ammonium hydroxide for three

^{*}Specification for sulphuric acid (revised).

[†]Specification for nitric acid.

Modified Gutzeit method of test for arsenic.

minutes followed by washing five times with water. Then tone the paper by dipping in auric chloride solution for five minutes. Finally wash the paper five times with water and then note whether any violet colouration has developed on the sensitized mercuric bromide paper.

B-4.2.1 The material shall be considered to be free from antimony if no violet colouration has developed on the sensitized mercuric bromide paper.

APPENDIX C

(*Clause* 5.1)

SAMPLING OF AMMONIUM CHLORIDE, TECHNICAL AND PURE

C-1. GENERAL REQUIREMENTS OF SAMPLING

C-1.0 In drawing, preparing, storing and handling samples, the following precautions and directions shall be observed.

C-1.1 Samples shall not be taken at a place exposed to weather.

C-1.2 The sampling instrument shall be clean and dry.

C-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

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

C-1.5 The samples shall be placed in clean, dry and air-tight glass or other suitable containers on which the material has no action.

C-1.6 The sample containers shall be of such a size that they are almost completely filled with the sample.

C-1.7 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling, month and year of manufacture, and other important particulars of the consignment.

C-2. SCALE OF SAMPLING

C-2.1 Lot — All the containers in a single consignment of the material of the same grade and type, drawn from a single batch of manufacture, shall constitute a lot. If a consignment is declared to consist of different

batches of manufacture, the batches shall be marked separately and the group of containers in each batch shall constitute separate lots. In the case of a consignment drawn from a continuous process, 1000 containers (or 100 tonnes) of the material shall constitute a lot.

C-2.2 The number of containers to be chosen from a lot shall depend on the size of the lot and shall be as given in Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

LOT SIZE	NUMBER OF CONTAINERS TO BE SELECTED
N	n
(1)	(2)
Up to 100	б
101,, 300	6
301 500	7
501 800	8
801 1300	9
1 301 and above	10

C-2.3 These containers shall be chosen at random from the lot, and in order to ensure randomness of selection a random number table as agreed to between the purchaser and the supplier shall be used. In case such a table is not available, the following procedure may be adopted:

Arrange all the containers in the lot in a systematic manner and starting from any container, count them as 1, 2, 3.......... up to r and so on, r being equal to the integral part of N/n. Every rth container thus counted shall be withdrawn and all such containers shall constitute the sample.

C-3. TEST SAMPLES AND REFEREE SAMPLE

C-3.1 Draw with an appropriate sampling instrument small portions of the material from different parts of the containers selected, the total quantity taken out from each container being sufficient to conduct the tests for all characteristics given in 3.

C-3.2 Mix thoroughly all portions of the material drawn from the same container to form an individual test sample. Equal quantities from all individual test samples so formed shall be mixed together to form a composite test sample.

C-3.3 All the individual test samples and the composite test sample shall be divided into three equal parts, thus forming three sets of test samples. These parts shall be immediately transferred to thoroughly dried bottles

which shall then be sealed air-tight with glass stoppers. These shall be labelled with all the particulars of sampling given under C-1.7. One of these sets of test samples shall be sent to the purchaser and another to the supplier.

C-3.4 Referee Sample — The third set of test samples, bearing the seals of the purchaser and the supplier, shall constitute the referee sample and shall be used in case of dispute between the purchaser and the supplier. It shall be kept at a place agreed to between the purchaser and the supplier.

C-4. NUMBER OF TESTS

C-4.1 Test for the determination of ammonium chloride shall be conducted on the each of the individual test samples.

C-4.2 Tests for the remaining characteristics given in 3 shall be conducted on the composite test sample.

C-5. CRITERIA FOR CONFORMITY

C-5.1 For Individual Samples—From the test results for ammonium chloride, the mean (x) and range (R) shall be calculated (range being defined as the difference between the maximum and the minimum of the test results).

C-5.2 The lot shall be declared as conforming to the requirement of ammonium chloride if the value of the expression (x - 0.6 R) is greater than or equal to 98.5 for technical grade and 99.5 for pure grade.

C-5.3 For Composite Sample—For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite sample, the test result for each of the characteristics shall satisfy the relevant requirement specified in Table 1.

BUREAU OF INDIAN STANDARDS

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

Centre Laboratory:	anaksanstha to all Offices)
Central Laboratory .	
Plot No. 20/9, Sile IV, Sanibabad Industrial Area, Sanibabad 201010	0-11 00 32
Regional Unices:	000 70 17
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
Piot No. 62-63, Unit VI, Ganga Nagar, BHUBANESHWAR 751001	40 36 27
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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
5-8-56C, 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
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NIT BUilding, Second Floor, Gokulpat Market, NAGPUR 440010	52 51 71
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Institution of Engineers (India) Building 1332 Shivaji Nagar, PUNE 411005	32 36 35
T.C. No. 14/1421, University P. O. Palayam, THIRUVANANTHAPURAM 695034	6 21 17
*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

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AMENDMENT NO. 2 JULY 1983

TO

IS:1113-1965 SPECIFICATION FOR AMMONIUM CHLORIDE, TECHNICAL AND PURE

(Revised)

Alterations

(Pages 13 and 14, clauses B-4 to B-4.2.1) -Substitute the following for the existing clauses:

'B-4. TEST FOR ANTIMONY

B-4.1 Reagents

B-4.1.1 Dilute Sulphuric Acid - 1 : 3(v/v).

B-4.1.2 Potassium Iodide Solution - 1 N.

B-4.1.3 Benzene

B-4.1.4 Rhodamine BS 100 - 0.2 percent solution.

B-4.2 Procedure - To test the solution of ammonium chloride, add 5 drops of dilute sulfuric acid and one drop of potassium iodide and 1 ml of benzene. Shake the mixture vigorously to extract antimony into the benzene layer. On a spot plate transfer 4 to 6 drops of the benzene layer (benzene extract) and add one drop of rhodamine BS 100. A violet benzene layer indicates the presence of antimony.'

(CDC 56)