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भारतीय मानक मसौदा

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(आइ एस 1114 का दूसरा पुनरीक्षण)

Draft Indian Standard

AMMONIUM CHLORIDE, FERTILIZER GRADE – SPECIFICATION

(Second Revision of IS 1114)

ICS No. 65.080

Soil Quality and Fertilizers Sectional
Committee, FAD 07

Last Date of Comments: 1 July 2024

FOREWORD

(Formal clauses would be added later)

This standard was first published in 1957 and covered the material used as a fertilizer as well as that for other purposes. The Sectional Committee responsible for the preparation of this standard reviewed the requirements prescribed in this specification vis-a-vis IS 1113 : 1965 'Specification for ammonium chloride, technical and pure (*revised*)', and felt that the material other than that used as a fertilizer was adequately covered by IS 1113. The Committee, therefore, decided to confine this specification to the material used as fertilizer only and the title of the specification was modified through first revision issued in 1964.

In this second revision, the standard has been aligned with Fertilizer Control Order, 1985 and the following major changes have been made:

- a) The volumetric method used for determination of ammoniacal nitrogen has been incorporated as an alternate method.
- b) The method used for determination of chlorides other than ammonium chloride has been modified.

In the preparation of this standard, due consideration has been given to the *Fertilizer Control Order*, 1985. However, this standard is subject to restrictions imposed under these, wherever applicable.

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 : 2022 'Rules for rounding off numerical values (*second*

revision)'. 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

This standard prescribes the requirements and the methods of sampling and test for ammonium chloride, fertilizer grade.

2 REFERENCES

The standards listed below contain provisions which through reference in this text, constitute provisions 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 listed below:

<i>IS No.</i>	<i>Title</i>
IS 264 : 2005	Nitric acid – Specification (<i>third revision</i>)
IS 1070 : 2023	Reagent grade water – Specification (<i>fourth revision</i>)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of white crystals or granules or powder, free from hard caking, and shall have no perceptible odour.

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

Table 1 Requirements for Triple Superphosphate, Fertilizer Grade
(*Clause 3.2*)

Sl. No.	Characteristic	Requirement	Method of test (Ref to Annex A)
(1)	(2)	(3)	(4)
i)	Moisture, percent by mass, <i>Max</i>	2.0	A-2
ii)	Ammoniacal nitrogen (as N), percent by weight, <i>Min</i>	25.0	A-3
iii)	Chlorides other than ammonium chloride (as NaCl), percent by weight on dry basis, <i>Max</i>	2.0	A-4

4 PACKING

The material shall be packed in moisture-proof multi-wall paper, or jute bags lined with polyethylene films, or in such other containers as agreed to between the purchaser and the supplier.

5 MARKING

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

- a) Name and address of manufacturer
- b) Trade-mark and/or brand name, if any;
- c) Name of the fertilizer, in case of imported fertilizer the word 'Imported' shall be superscribed;

- d) Percent nutrient as total nitrogen to be denoted by the letter N;
- e) Gross and net quantity in kilogram, when packed;
- f) Any other requirements as specified under the *Fertilizer Control Order*, 1985 and the *Legal Metrology (Packaged Commodities) Rules*, 2011.

5.2 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.

6 SAMPLING

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

7 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.

ANNEX A
(Clause 3.2, Table 1)
ANALYSIS OF AMMONIUM CHLORIDE, FERTILIZER GRADE

A-1 PREPARED SAMPLE

Mix the sample well by swirling 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 sample. Do not expose the sample to an atmosphere containing acid or alkaline fumes.

A-2 DETERMINATION OF MOISTURE

A-2.1 Procedure

Weigh accurately about 5 g of the prepared sample (*see A-1*) in a weighed shallow porcelain dish and dry for 24 h in a vacuum desiccator over sulphuric acid and reweigh. Preserve the dried material for subsequent tests.

A-2.2 Calculation

$$\text{Moisture, percent by weight} = \frac{M_1}{M_2} \times 100$$

where,

M_1 = loss in mass in g on drying, and

M_2 = mass in g of the prepared sample (*see A-1*) taken for the test.

A-3 DETERMINATION OF AMMONIACAL NITROGEN

A-3.1 Two methods have been specified for determination of ammoniacal nitrogen in ammonium chloride. Any one may be used depending on the facilities available.

A-3.2 Method I

A-3.2.1 Apparatus

The apparatus, as assembled, is shown in Fig. 1. It consists of a flask *A* of 1000 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.

A-3.2.2 Reagents

A-3.2.2.1 *Standard sulphuric acid* – 0.1 N.

A-3.2.2.2 *Methyl red indicator* - Dissolve 0.03 g of methyl red in 100 ml of water.

A-3.2.2.3 *Magnesium oxide* – Freshly ignited and free from carbonate.

A-3.2.2.4 *Standard sodium hydroxide solution* – 0.1 N.

A-3.2.3 Procedure

Powder the prepared sample (*see A-1*) finely in a clean dry glass or porcelain pestle and mortar. Weigh accurately about 0.2 g of it 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 50 ml of standard sulphuric acid contained in the beaker *E* to which a few drops of methyl red indicator have been added. Add about 5 g of freshly ignited magnesium oxide to the flask *A* 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 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.

Carry out a blank test using all reagents in the same quantities but without the prepared sample (*see A-1*).

A-3.2.4 Calculation

$$\text{Ammoniacal nitrogen (as N), percent by mass} = \frac{1.4008 (B-A) N}{M}$$

where,

B = volume in ml of standard sodium hydroxide solution used to neutralize the acid in the blank determination;

A = volume in ml of standard sodium hydroxide solution used to neutralize the excess of acid in the test with the material;

N = normality of standard sodium hydroxide solution; and

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

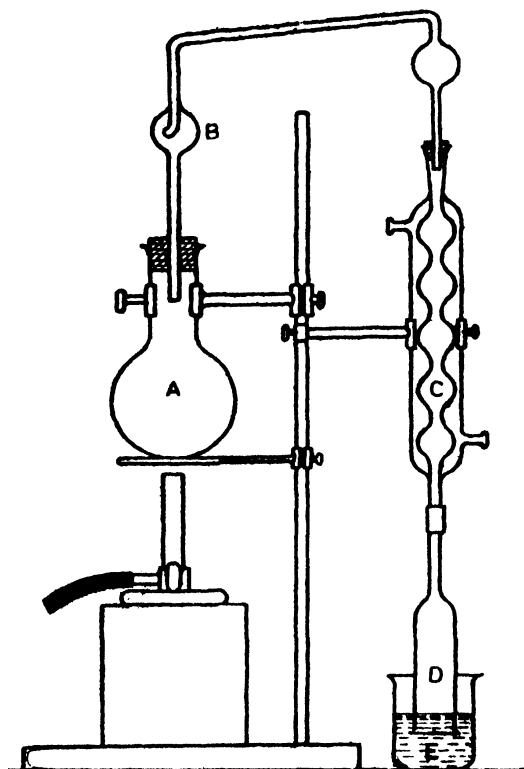


FIG 1 APPARATUS FOR THE DETERMINATION OF AMMONIACAL NITROGEN

A-3.3 Method II

A-3.3.1 Procedure

Two procedures have been specified for determination of ammoniacal nitrogen in ammonium chloride depending on whether the fertilizers contain urea or not.

A-3.3.1.1 Procedure I – Fertilizers with no urea

Dissolve 1 g prepared sample in 250 ml water. Take 25 ml aliquot in 1 litre distillation flask, add approximately 300 ml of water. Add 10 ml of 45% NaOH for fertilizers which do not contain urea. Immediately connect the distillation flask to the distillation assembly and swirl to mix the contents. Heat until all ammonia is distilled (at least 150 ml distillate). Test with red litmus paper, if any ammonia is still coming out. Remove receiving flask. Rinse outlet tube in to receiving flask with a small amount of distilled water. Titrate the contents in the receiver conical flask with 0.1 N NaOH. Determine blank on reagents using same quality of standard acid in receiver conical flask.

A-3.3.1.2 Procedure II – Fertilizers with urea

Add 2 g of freshly ignited carbonate free MgO for fertilizers containing urea. Immediately connect the distillation flask to the distillation assembly and swirl to mix the contents. Heat until all ammonia is distilled (at least 150 ml distillate). Test with red litmus paper, if any ammonia is still coming out. Remove receiving flask. Rinse outlet tube in to receiving flask with a small amount of distilled water. Titrate the contents in the receiver conical flask with 0.1 N NaOH. Determine blank on reagents using same quality of standard acid in receiver conical flask.

A-3.3.2 Calculations

$$\text{Nitrogen per cent (by weight)} = \frac{1.401 (V_1 N_1 - V_2 N_2) - (V_3 N_1 - V_4 N_2) \times F}{M}$$

where,

V_1 = Volume (in ml) of standard acid taken in receiver flask for sample.

V_2 = Volume (in ml) of standard NaOH used in titrating standard acid in receiver flask after distillation.

V_3 = Volume (in ml) of standard acid taken in receiver flask for blank.

V_4 = Volume (in ml) of standard NaOH used in titrating standard acid in receiver flask after distillation in blank.

N_1 = Normality of standard acid.

N_2 = Normality of standard NaOH.

M = mass in g of sample taken.

F = Dilution factor of the sample.

A-4 DETERMINATION OF CHLORIDES OTHER THAN AMMONIUM CHLORIDE

A-4.1 Reagents

A-4.1.1 *Standard Silver Nitrate Solution* — 0.1 N.

A-4.1.2 *Concentrated Nitric Acid* — conforming to IS 264.

A-4.1.3 *Ferric Ammonium Sulphate Solution* — saturated in water and stabilized by addition of 50 ml nitric acid.

A-4.1.4 *Standard Ammonium Thiocyanate Solution* — 0.1 N.

A-4.2 Procedure

Dissolve about 0.2 g, of the prepared sample (*see A-1*), previously dried as in procedure for determination of moisture and accurately weighed, in about 40 ml water. Add exactly 50 ml of standard silver nitrate solution and 5 ml of concentrated nitric acid. Add 0.5 ml of nitrobenzene and make up the volume of the mixture to exactly 100 ml with water. Take exactly 50 ml of the solution and add 2 ml of ferric ammonium sulphate solution. Titrate the excess of silver nitrate in this portion with standard ammonium thiocyanate solution.

Carry out a blank test following the procedure given as above but without using the material

A-4.3 Calculation

A-4.3.1 Total chlorides (Cl), percent by mass (on dry basis) = $\frac{7.094 (V_1 - V_2) \times N}{M}$

where,

V_1 = volume in ml of standard ammonium thiocyanate solution used in the blank determination,

V_2 = volume in ml of standard ammonium thiocyanate solution used in the test with the material,

N = normality of standard ammonium thiocyanate solution, and

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

A-4.3.2 Express the ammoniacal nitrogen content, percent by mass, of the material as determined in **A-3** in terms of chloride as follows:

A-4.3.2.1 Chloride equivalent of the ammoniacal nitrogen content, percent by mass = $2.531 \times A$

where,

A = ammoniacal nitrogen content determined in **A-3**

A-4.3.2.2 Balanced chloride equivalent to sodium chloride (NaCl), percent by mass = $1.648 \times (X - Y)$

where,

$(X - Y)$ = Balanced chlorides other than ammonium chloride.

ANNEX B

(Clause 6)

SAMPLING OF AMMONIUM CHLORIDE, FERTILIZER GRADE

B-1 GENERAL REQUIREMENTS OF SAMPLING

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

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

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

B-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.

B-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.

B-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.

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

B-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.

B-2 SCALE OF SAMPLING

B-2.1 Lot

All the containers in a single consignment of the material 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 groups of containers in each batch shall constitute separate lots. In the case of a consignment drawn from a continuous process, 1 000 containers (or 100 tonnes) of the material shall constitute a lot.

B-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.

Lot Size	No. of Containers to be Selected
(1)	(2)
N	n
Upto 100	5
101 to 300	6
301 to 500	7
501 to 800	8
801 to 1300	9
1301 and above	10

B-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:

Starting from any container, count them as 1, 2, 3, up to r and so on in one order. Every r^{th} container thus counted shall be withdrawn and used in the preparation of a composite sample, where r is the integral part of N/n .

B-3 TEST SAMPLES AND REFEREE SAMPLE

B-3.1 From each of the containers selected according to **B-2.2** and **B-2.3**, an equal quantity of the material shall be taken and thoroughly mixed to constitute a composite sample weighing not less than 100 g.

B-3.2 This composite test sample shall be divided into three equal parts, thus forming three test samples. These shall be immediately transferred to thoroughly dried bottles which shall then be sealed air-tight with glass stopper. These shall be labelled with all the particulars of sampling given under **B-1.7**. One of these test samples shall be sent to the purchaser and another to the supplier.

B-3.3 Referee Sample - The third test sample, 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.

B-4 NUMBER OF TESTS

Tests for all the characteristics shall be conducted on the composite sample.

B-5 CRITERIA FOR CONFORMITY

A lot shall be declared as conforming to the requirements of this specification if the test results for all the characteristics satisfy the relevant requirements given in this specification