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IS 12478 (1988): Ammonium sulphate, technical [CHD 1: Inorganic Chemicals]



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Indian Standard
SPECIFICATION FOR
AMMONIUM SULPHATE, TECHNICAL

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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard

SPECIFICATION FOR AMMONIUM SULPHATE, TECHNICAL

0. FOREWORD

0.1 This Indian Standard was adopted by the Bureau of Indian Standards on 22 August 1988, after the draft finalized by the Inorganic Chemicals (Miscellaneous) Sectional Committee had been approved by the Chemical Division Council.

0.2 Apart from its use as nitrogenous fertilizer (see IS : 826-1980*), ammonium sulphate is also used in the treatment of hides and skin for leather production. In combination with phosphate and borate, it is used for fire proofing timber, plastics and insulating materials. In combination with chlorine, ammonium sulphate disinfects water. It is also used as a nutrient,

*Specification for ammonium sulphate, fertilizer grade (second revision).

in yeast production from molasses and in the manufacture of explosives and ammunition.

0.3 This standard, however, covers the requirements for ammonium sulphate used as a water treatment chemical, in the treatment of hides and skins and in the manufacture of explosives and ammunition.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Rules for rounding off numerical values (revised).

1. SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for ammonium sulphate, technical.

2. TYPES

2.1 The material shall be of the following three types;

- a) Type 1 — for leather processing,
- b) Type 2 — for water treatment, and
- c) Type 3 — for explosives and ammunition.

3. REQUIREMENTS

3.1 Description — The material shall be in the form of free flowing crystal or powder and shall be free from extraneous material.

3.2 The material shall also comply with the requirements given in Table 1 when tested according to methods given 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 in jute or multi-walled paper bags or jute lined with polyethylene film or in such other containers as agreed to between the purchaser and the supplier. Each bag shall be securely closed.

4.2 Marking — Each bag shall bear legibly and indelibly the following information:

- a) Name and type of the material;
- b) Name of the manufacturer and his recognized trade-mark, if any;
- c) Percentage of assay by mass;
- d) Gross and net mass in kg; and
- e) Batch number.

4.2.1 The container 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 Representative test samples of the material shall be drawn as prescribed in IS : 6092 (Part 1)-1985*.

*Methods of sampling and test for fertilizers: Part 1 Sampling (first revision).

TABLE 1 REQUIREMENTS FOR AMMONIUM SULPHATE, TECHNICAL

(Clauses 3.2 and 5.2)

Sl No.	CHARACTERISTICS	REQUIREMENTS			METHOD OF TEST, REFERENCE TO CLAUSE No. OF APPENDIX A
		Type 1	Type 2	Type 3	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Assay, percent by mass, <i>Min</i>	96.7	98.0	99.0	A-3
ii)	Moisture, percent by mass, <i>Max</i>	1.0	0.25	0.25	A-4
iii)	Free acidity (as H_2SO_4), percent by mass, <i>Max</i>	0.70	0.15	0.025	A-5
iv)	Water insolubles, percent by mass, <i>Max</i>	To pass the test	—	0.05	A-6
v)	Iron (as Fe), percent by mass, <i>Max</i>	0.20	—	—	A-7
vi)	Ether soluble matter, percent by mass, <i>Max</i>	—	0.03	—	A-8
vii)	Arsenic, percent by mass, <i>Max</i>	0.01	0.005	0.05	A-9
viii)	Sulphated ash, percent by mass, <i>Max</i>	—	—	0.40	A-10
ix)	Total chlorides (as NH_4Cl), percent by mass, <i>Max</i>	—	—	0.05	A-11
x)	Nitrates (as NO_3), percent by mass, <i>Max</i>	—	—	Nil	A-12

5.2 Criteria for Conformity — For declaring the conformity of the lot to the requirements of this specification, the test results on the composition sample shall satisfy all the requirements specified in Table 1.

APPENDIX A

(Clause 3.2)

METHODS OF TEST FOR AMMONIUM SULPHATE, TECHNICAL

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (see IS : 1070-1977*) shall be used in tests.

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

A-2. PREPARATION OF SAMPLE FOR ANALYSIS

A-2.1 Procedure — Reduce the test sample to a quantity sufficient for analysis and grind not less than 0.255 kg of reduced sample without previous sieving. For materials that form a paste on putting pressure, grind in porcelain pestle and mortar (see Note) to pass through 1 mm IS Sieve. For dry mixtures that tend to segregate, grind in a porcelain pestle and mortar (see Note) to pass through 355 microns IS Sieve.

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

Grind as rapidly as possible to avoid loss or gain of moisture during operation. Mix thoroughly and store in tightly stoppered bottles of sizes that are almost completely filled by the ground sample.

NOTE — For hygroscopic samples, it is preferable to use electric mixer.

A-3. ASSAY

A-3.1 Reagents

A-3.1.1 Formaldehyde Solution — Neutralize 37 percent formaldehyde solution with 0.25 N sodium hydroxide to phenolphthalein end point. Filter in case some precipitates have formed.

A-3.1.2 Phenolphthalein Indicator — 0.5 percent (*m/v*) solution in 50 percent ethyl alcohol.

A-3.1.3 Standard Sodium Hydroxide Solution — 1 N (see IS : 2316-1968*).

*Methods of preparation of standard solutions for colorimetric and volumetric analysis (first revision).

A-3.2 Procedure — Dissolve about 2.5 g of the material in water and add a mixture of formaldehyde solution and 25 ml of water. Allow to stand for 30 minutes and titrate against 1 N sodium hydroxide solution to the phenolphthalein end point.

A-3.3 Calculation

$$\text{Assay [as } (\text{NH}_4)_2\text{SO}_4 \text{], percent by mass} \\ = \frac{66}{1000} \times \frac{V \times N \times 100}{M}$$

where

V = volume in ml of standard sodium hydroxide required for the titration,

N = normality of standard sodium hydroxide solution, and

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

A-4. MOISTURE

A-4.1 Procedure — Weigh to the nearest milligram about 2 g of the prepared sample in a weighed, clean, dry squat form weighing bottle. Heat in an air oven for about 5 hours at 129 to 131°C to constant mass. Cool in a desiccator and weigh.

A-4.2 Calculation

$$\text{Moisture, percent by mass} = \frac{100 (M_1 - M_2)}{M_1 - M_3}$$

where

M_1 = mass in g of the bottle with the material before drying,

M_2 = mass in g of the bottle with the material after drying, and

M_3 = mass in g of the bottle.

A-5. FREE ACIDITY

A-5.1 Reagents

A-5.1.1 Standard Sodium Hydroxide Solution — 0.02 N.

A-5.1.2 Methyl Red Indicator — Dissolve 0.2 g of methyl red indicator in 100 ml neutral rectified spirit.

A-5.1.3 Methyl Red — Methylene Blue Mixed Indicator Solution — Prepare by mixing equal volumes of 0.2 percent solution of methyl red and 0.1 percent solution in rectified spirit of methylene blue.

A-5.2 Procedure — Dissolve about 5 g of the prepared sample, accurately weighed, in about 50 ml of cold water. Filter (see Note), if necessary, and make up the volume to about 200 ml. Titrate with standard sodium hydroxide solution, using one or two drops of methyl red as indicator. If satisfactory end point with methyl

red is not obtained, methyl red-methylene blue mixed indicator may be used. Use preferably a microburette for this titration.

NOTE — The filtering medium shall be neutral and shall not contain any alkaline material which would neutralize the free acid.

A-5.3 Calculation

$$\text{Free acidity (as H}_2\text{SO}_4 \text{), percent by mass} \\ = \frac{4904 V N}{M}$$

where

V = volume in ml of standard sodium hydroxide solution,

N = normality of standard sodium hydroxide solution, and

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

A-6. WATER INSOLUBLES

A-6.1 Apparatus

A-6.1.1 Gooch Crucible — G. No. 4.

A-6.1.2 Oven — maintained at $100 \pm 2^\circ\text{C}$.

A-6.1.3 Muffle Furnace — maintained at $800 \pm 25^\circ\text{C}$.

A-6.2 Procedure

A-6.2.1 Take 5 g of the material in a 500 ml beaker and dissolve in about 100 ml of water and stir thoroughly (heat to boiling, if necessary). Cover the beaker with a watch glass and allow it to stand for 4 hours at room temperature. Filter the residue in a weighed Gooch crucible. Apply suction and wash the residue with water. Dry the crucible in an oven maintained at $100 \pm 2^\circ\text{C}$ till constant mass is obtained. Weigh the crucible with residue and find out the total matter insoluble in water.

A-6.3 Calculation

a) Water insolubles, percent by mass

$$= \frac{(M_2 - M_1) \times 100}{M}$$

where

M = mass in g of the sample taken for the test,

M_1 = mass in g of empty Gooch crucible, and

M_2 = mass in g of Gooch crucible and water insolubles.

A-7. IRON

A-7.1 Reagents

A-7.1.1 Nitric Acid — See IS : 264-1976*.

*Specification for nitric acid (second revision).

A-7.1.2 Hydrochloric Acid — See IS : 265-1987*.

A-7.1.3 Ammonia

A-7.1.4 Ammonium Chloride

A-7.2 Procedure — Weigh accurately about 10 g of the material and dissolve in 10 ml nitric acid and 100 ml of water by heating. Precipitate iron by adding ammonia till the colour of the solution turns deep blue, heat to boil and filter. Wash with dilute solution of ammonia till the washings are colourless. Dissolve the residue in 10 ml of hydrochloric acid and 50 ml of water taken in a beaker. Add 5 g of ammonium chloride and stir to dissolve. Add 20 ml of ammonia, boil, cool and filter. Wash the precipitate with water, dry the residue, ignite, and weigh to constant mass.

A-7.3 Calculation

$$\text{Iron content, percent by mass} = \frac{M_1 \times 70}{M_2}$$

where

M_1 = mass in g of the residue, and

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

NOTE — Atomic absorption spectrometric method may be used as an alternative method.

A-8. ETHER SOLUBLE MATTER

A-8.1 Reagent

A-8.1.1 Ethyl Ether — anhydrous.

A-8.2 Procedure — Weigh accurately about 25 g of the sample after drying for 2 h at $100 \pm 2^\circ\text{C}$. Mix it with about 100 ml of ethyl ether in a beaker. Shake for about 30 minutes and filter. Take the filtrate in a tared porcelain dish, keep in an oven maintained at $100 \pm 2^\circ\text{C}$ and weigh to constant mass.

A-8.3 Calculation

Ether soluble matter, percent by mass

$$= \frac{M_1}{M_2} \times 100$$

where

M_1 = mass in g of ether soluble residue, and

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

A-9. ARSENIC

A-9.1 Procedure — Dissolve 10.0 g of the material in about 30 ml of water and carry out the test as prescribed in IS : 2088-1983†.

*Specification for hydrochloric acid (third revision).

†Methods for determination of arsenic (second revision).

A-9.1.1 The material shall be considered to be free from arsenic if no stain is produced on the sensitized mercuric bromide paper.

NOTE — Atomic absorption spectrophotometric method may be adopted as an alternative method.

A-10. SULPHATED ASH

A-10.1 Reagents

A-10.1.1 Concentrated Nitric Acid — See IS : 264-1976*.

A-10.1.2 Concentrated Sulphuric Acid — See IS : 266-1977†.

A-10.2 Procedure — Heat the crucible with a small bunsen flame muffle furnace for 15 minutes and cool it to room temperature in a desiccator and weigh. Place about 5 g of the sample in the weighed crucible and weigh again. Heat the crucible steadily with a small bunsen flame. Burn off the residual carbon to a dull red hot and then allow it to cool at room temperature in open. Treat the ash with a few drops of concentrated nitric acid followed by a few drops of concentrated sulphuric acid and again heat in fuming chamber until the acids have volatilized and the whole of the carbon has burnt off. Cool the crucible in desiccator and weigh again (M_2).

A-10.3 Calculation

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

where

M = mass in g of the empty crucible,

M_1 = mass in g of the crucible and sample, and

M_2 = mass in g of the crucible and ash.

A-11. TOTAL CHLORIDES

A-11.1 Reagents

A-11.1.1 Sodium Carbonate — powder.

A-11.1.2 Dilute Nitric Acid — 1 : 4.

A-11.1.3 Potassium Chromate Solution — saturated.

A-11.1.4 Standard Silver Nitrate Solution — N/20.

A-11.2 Procedure — Take about 5 g of the sample in a conical flask. Add to the flask, 100 ml of fresh water. Shake the solution and filter through No. 40 Whatman filter paper. Wash the residue twice using about 5 to 10 ml of water. Transfer the solution to a porcelain dish

*Specification for nitric acid (second revision).

†Specification for sulphuric acid (second revision).

and make neutral to phenolphthalein either with sodium carbonate (Na_2CO_3) or with dilute nitric acid as appropriate. Add two drops of saturated solution of potassium chromate (free from chloride). Titrate with N/20 standard solution of silver nitrate until a faint but standard permanent reddish tinge appears which indicates that all the chloride has combined with silver and the slight excess has formed a precipitate of silver chromate (Ag_2CrO_4).

11.2.1 Carry out blank using all the reagents and apply the necessary correction.

A-11.3 Calculation

$$\text{Total chlorides (as } \text{NH}_4\text{Cl),} \\ \text{percent by mass} = \frac{5.35 V N}{M}$$

where

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

N = normality of the standard silver nitrate solution used, and

M = mass of the sample taken.

A-12. NITRATES

A-12.1 Apparatus

A-12.1.1 *White Porcelain Tile* — for spot test.

A-12.1.2 *Pointed Glass Rod*

A-12.2 Reagent

A-12.2.1 *Diphenylamine* — 1 percent solution in concentrated sulphuric acid.

A-12.3 Procedure — Dissolve 0.5 g of the sample in about 100 ml of water followed by 50 mg of sodium chloride. When the latter has dissolved, add 0.2 ml of diphenylamine solution. To this, add without agitation 12 ml concentrated sulphuric acid. After a few minutes, mix gently and allow it to stand for 30 minutes. No blue colour should appear.

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