

PAKISTAN STANDARD

SPECIFICATION FOR AMMONIUM SULPHATE (FERTILIZER)



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PAKISTAN STANDARDS AND QUALITY CONTROL AUTHORITY,
STANDARDS DEVELOPMENT CENTRE,
PSQCA COMPLEX, PLOT NO. ST – 7/A, BLOCK NO. 3,
SCHEME – 36, GULISTAN-E-JAUHAR,
KARACHI.

0. FOREWARD

- 0.1 This Pakistan Standard (2nd Revision) was adopted by Pakistan Standards Quality Control Authority on 26-11-2014, after the draft finalized by the Fertilizers and Allied Product Technical Committee had been approved by the National Standards Committee for Chemical.
- 0.2 The Pakistan Standard Specification was first established in 1958 and first revised in 1975, keeping in view the latest developments made in the industries; the committee felt it is necessary to further revise, update and harmonizing the Pakistan Standard with International Standards.
- 0.3 In the preparation of this standard, assistance has been derived from IS: 826- 1980 (Rev.) and SLS: 620-1987 acknowledged with thanks. A basic requirement of the material in laid down in this standard, to assist manufactures, users and testing authority.
- 0.4 While preparing this standard the views of the Manufacturers, importers, testing authorities, Technologist/ experts, all stakeholders and consumers, have been taken into consideration and also the existing trade practice in this field in the country, by the Technical Committee. Further more, due weightage had to be given to the need for international co-ordination among standard prevailing in different countries of the world.
- 0.5 This standard is intended mainly to cover the technical provisions relating to the supply of the material, and it does not cover all the necessary provision of a contract.
- 0.6 In order to keep abreast with the progress of trade and Industry Pakistan Standards are revise periodically. Suggestions from the members are welcomed and will be placed before the Technical Committees for consideration at the time of revision.
- 0.7 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 PS: 103 – 1991 “Rules for Rounding Off Numerical Values”. The number of places retained in the rounded off value should be the same as those of the specified value in the standard.

1. SCOPE

- 1.1 This Pakistan Standard prescribed the requirements, the methods for sampling and test for ammonium sulphate, fertilizer grade. The material may also be used for other industrial purpose.

2. REQUIREMENT:

- 2.1 **Description:** The material shall be in the form of free-flowing crystals, and shall be free from extraneous material.
- 2.2 The material shall also comply with the requirements given in Table 1.

TABLE-1

Requirements For Ammonium Sulphate, Fertilizer Grade

S.#	Characteristics	Requirements	Method of Tests Ref Part & Cl. No.
(1)	(2)	(3)	(4)
i)	Moisture, percent by mass, Max	1.0	B-3
ii)	Ammonical Nitrogen, percent by mass, Min	20.6	B-4
iii)	Free acidity (as H ₂ SO ₄), percent by mass, Max	0.025	B-5
iv)	Arsenic (as AS ₂ O ₃), ppm, Max	0.50	B-6
<p>* <i>Methods of sampling and test for fertilizers:</i> <i>Part II Determination of nitrogen.</i> <i>Part VI Determination of impurities</i> <i>0.04 for material obtained from by-product ammonia and by-product gypsum</i> * <i>Specification for test sieves: Part I Wire cloth test sieve (2nd Rev) Rules for rounding off numerical values (revised).</i></p>			

3.0 PACKING AND MARKING

3.1 **Packing:** - The material shall be packed in natural / synthetic fiber bags lined with and multiwall paper or a suitable plastic material or in the suitable containers as agreed between the purchaser and the vendor. The weight of the material in a bag shall ordinarily be 50 kg.

3.2. **Marking-** The container/ sack shall be securely closed and marked with the following information:

- (a) Name of the material, namely "Ammonium Sulphate" fertilizer grade
- (b) The Percentage of ammonical Nitrogen content of the material
- (c) Name and Address of the manufacturer/importer/distributor; trade mark if any
- (d) Net Weight in kg of the material in the bag, and
- (e) Each bag may also be marked with PSQCA certification
- (f) Any information required by law enforcement agencies or by the buyer.

4.0 SAMPLING

4.1 Representative sample of the material shall be drawn as prescribed in Appendix A.

APPENDIX – A

SAMPLING OF AMMONIUM SULPHATE (FERTILIZER GRADE)

A-1 GENERAL REQUIREMENTS OF SAMPLING

- A-1.0 In drawing preparing, storing and handling test samples, the following precautions and directions shall be observed.
- A-1.1 Sampling shall be taken at a place protected from damp air, dust and soot.
- A-1.2 The sampling instruments shall be clean and dry when used.
- A-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instruments and the containers for samples from adventitious contamination.
- A-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.
- A-1.5 The samples shall be placed in clean, dry and airtight glass or other suitable containers on which the material has no action.
- A-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.
- A-1.7 Each sample container shall be sealed airtight after filling and marked with full details of sampling, the date of sampling and other important particulars of the consignment.
- A-1.8 Samples shall be stored in a cool and dry place.

A-2 **SCALE OF SAMPLING:**

- A-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 such batch shall constitute separate lots. In the case of consignment drawn from a continuous process, 1,000 containers (or 100 metric tons of the material) shall constitute a lot.
- A-2.2 The number of containers to be chosen from a lot shall depend on the size of the lot and shall be in accordance with column 1 and 2 of Table II.

TABLE – II
NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

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

- A-2.3 These containers shall be chosen at random from the lot, and in order to ensure randomness of selection the following procedure may be adopted.
- A-2.4 Arrange all the containers in the lot in a systematic manner and starting from any container, count them 1,2,3, etc up to r and so on, r being equal to the integral part of N/n. Every rth containers thus counted shall be withdrawn and all such containers shall constitute the sample.

A-3 TEST SAMPLE AND REFEREE SAMPLE:

- A-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 table-1.
- A-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.
- A-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 stopper. One of these sets of test sample shall be sent to the purchaser and another to the vendor.
- A-3.4 **Referee Sample** – The third set of test samples bearing the seals of the purchaser and the vendor, shall constitute the referee sample and shall be used in case of dispute between the purchaser and the vendor. It shall be kept at a place agreed to between the purchaser and the vendor.

A-4 NUMBER OF TEST:

- A-4.1 Test for the determination of total nitrogen shall be conducted on each of the individual test samples.
- A-4.2 Test for the remaining characteristics given in table-1 shall be conducted on the composite test sample.

A-5 CRITERION FOR CONFORMITY:

- A-5.1 The test results for total nitrogen shall be recorded as shown in Table III. The mean and the range of the test result shall be calculated as follows:

$$\text{Mean } (\bar{X}) = \frac{\text{The sum of the test results}}{\text{Number of test results}}$$

Range (R) = the difference between the maximum and the minimum values of the test results.

- A-5.1.1 The appropriate expression as shown in col.6 of Table III shall be calculated for the characteristic. If the condition given in col.6 of Table III is satisfied, the lot shall be declared to have satisfied the requirement for this characteristic.
- A-5.2 For the remaining characteristics, the test results on the composite test sample shall satisfy the requirements specified in table-1.
- A-5.3 A lot shall be declared as conforming to the specification only when it has satisfied each of the requirements specified in table-1.

TABLE – III
CRITERION FOR CONFORMITY

S.#	Characteristic	Test Results	Mean	Range	Criterion for Conformity
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		1,2,..... n			
i.	ii.	iii	iv	v	vi
1	Total Ammonical nitrogen, % by weight	--	\bar{X}	R	$\bar{x}-0.6 R \geq$ the value specified in Table (1)

APPENDIX – B

B-0 ANALYSIS OF AMMONIUM SULPHATE, FERTILIZER GRADE

B-1 QUALITY OF REAGENTS:

B-1.1 Unless specified otherwise, recognized analytical reagent quality chemicals shall be employed in tests and distilled water freshly boiled and cooled, shall be used where the use of water as a reagent is intended.

B-2 PREPARATION OF SAMPLE:

B-2.1 Mix the sample well by rotating the bottle several times and transfer a portion immediately into a wide mouthed bottle and stopper it. Take care that no pieces of cork or sealing material get mixed with the sample. Do not expose the sample to an atmosphere containing acid or alkaline fumes. Use this prepared sample for test purpose.

B-3.0 DETERMINATION OF MOISTURE

B-3.1 **General** – A known weight of the material is dried at $105^{\circ} \pm 2^{\circ}\text{C}$ till constant weight is obtained.

B-3.1 **Procedure** – Weigh accurately 5.0 g of the prepared sample in a weighed, clean, dry squat from weighing bottle and dry in an oven at $105^{\circ} \pm 2^{\circ}\text{C}$. Cool in a desiccator and weigh. Repeat heating, cooling until constant weight is attained.

B-3 CALCULATION:

$$\text{Moisture, percent by weight} = 100 \times \frac{w}{W}$$

Where

w = loss in weight in g on heating and

W = weight in g of the prepared sample taken for the test.

B-4 DETERMINATION OF AMMONIACAL NITROGEN

B-4.1 **Apparatus** – A recommended, apparatus, as assembled is shown in Fig 1.

B-4.1.1 **Description** – The apparatus consist of the distillation 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.

- B-4.2 **Reagents:** - The following reagents are required,
- B-4.2.1 **Magnesium oxide** (Carbonate free) – Freshly ignited
- B-4.2.2 **Standard Sulphuric Acid**- Approximately 0.1 N
- B-4.2.3 **Standard Sodium Hydroxide Solution**- Approximately 0.1 N
- B-4.2.4 **Methyl Red Indicator**- Dissolve 1.0 g of methyl red in 200 mL of rectified spirit, 95 percent by volume

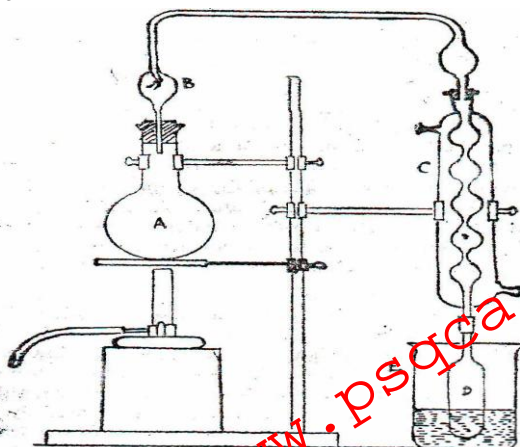


Fig 1. Assembly of Apparatus for the Determination of Ammoniacal Nitrogen

- B-4.3 **PROCEDURE:** - Weigh accurately about 5.0 g of the prepared sample and transfer in a beaker. Dissolve in water and filter. Collect the filtrate in a 500 mL volumetric flask, wash and make up the volume. Transfer 20 mL of the solution, corresponding to about 0.2 g of the sample, to the distillation flask. A and add about 500mL of water. Assemble the apparatus as shown in fig.1 with the tip of the condenser dipping in standard sulphuric acid in beaker E to which a few drops of methyl red indicator have been added.

Add about 10.0 g of freshly ignited magnesium oxide and distill until all ammonia has passed over. Lower the beaker E until the end of the dip tube D is out of the standard 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. Wash the dip tube D carefully so that all traces of condensate are removed. When all the washings have drained into the beaker E, add two or three drops of methyl red indicator and titrate with standard sodium hydroxide solution.

Carry out a blank using all reagents in the same quantities but without the material to be tested:

- B-4.4 **CALCULATION:**

$$\text{Ammonical nitrogen, percent by weight} = \frac{1.4008(B-A) N}{W}$$

Where,

B = Volume in mL of the standard sodium hydroxide solution used to neutralize the acid in blank determination,

A= Volume in mL of the standard sodium hydroxide solution used to neutralize the excess of acid in the test with the material,

W = weight in g of sample taken for nitrogen determination,

N= Normality of the standard sodium hydroxide solution.

B-5.0 DETERMINATION OF FREE ACIDITY (as H₂SO₄)

B-5.1 **Reagents:** - The following reagents are required,

B-5.1.1 **Standard Sodium Hydroxide Solution-** Approximately 0.02 N

B-5.1.2 **Methyl Orange** - Dissolve 0.50 g of methyl orange in water and dilute it to one litre.

B-5.2 **PROCEDURE:** - Dissolve about 20 g of the prepared sample accurately weighed, in about 50 mL of cold, neutral water. Filter and make up the volume to 200 mL. Titrate with standard sodium hydroxide solution using one or two drops of methyl orange. Use preferably a micro burette for this titration.

B-5.2.1 The filtering medium shall be neutral and shall not contain any alkaline material which would neutralize the free acid.

B-5.3 CALCULATION:

Free acidity (as H₂SO₄), percent by weight = $\frac{4.904 A N}{W}$

Where

A= Volume in mL of the standard sodium hydroxide solution

W = weight in gm of the prepared sample taken for the test.

N= Normality of the standard sodium hydroxide solution.

B-6 DETERMINATION OF ARSENIC IN FERTILIZERS

B-6.1 APPARATUS

- (i) Spectrophotometer with 50 mm cell.
- (ii) Volume flask : 25 ml, 500 ml, 1000 ml
- (iii) Burette : 25 ml
- (iv) Pipettes : 2 ml, 5 ml
- (v) Arsine generation & absorption apparatus

B-6.2 CHEMICAL/REAGENTS:

(i) **Pot. Iodide solution (15%)**

Dissolve 15g AR grade KI in 100 ml water

B-7.2 Stannous chloride Solution (40%)

Dissolve 4g SnCl₂ in 10ml conc. HCl and place it in dropper type indicator bottle.

B-7.3 HCl Conc.

B-7.4 Zinc Granules

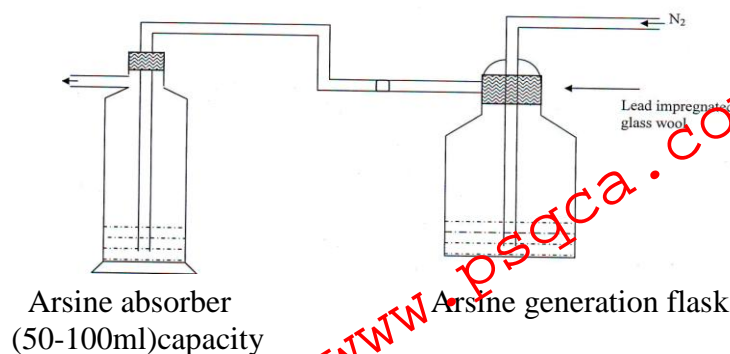
B-7.5 Lead Acetate Impregnated Glass Wool

Dissolve 10g lead acetate in 100ml water. Dip a piece of glass wool in this solution for few minutes dry with filter paper and complete dry at ambient temperature in desiccator.

- B-7.6 **Silver diethyl dithiocarbamate**
Dissolve 2.25g Sodium diethyl dithiocarbamate in 100 ml water. Dissolve 1.7g AgNO₃ in 100 ml water. Cool both solutions to 8-10°C. Add sodium diethyl dithiocarbamate solution to AgNO₃ solution. Yellow precipitate is formed. Filter and wash ppt. with cold water. Dry yellow cake in desiccator.
- B-7.7 **Silver diethyl dithiocarbamate Solution**
Dissolve 1.0 ml morpholine in 70ml chloroform (CHCl₃). Add 0.3g Silver diethyl dithiocarbamate, mix to dissolve and make up to 100ml with (CHCl₃). Store solution in refrigerator.
- B-7.8 **1000 ppm Standard Arsenic Solution**
Dissolve 1.734g sodium arsenite in water and make up to 1000ml, 1ml = 1 mg As.
- B-7.9 **1ppm Arsenic Standard Solution**
Dilute 25ml of 1000ppm solution to 500ml. 1ml = 50ug. Dilute 20ml of this solution to 1000ml. 1ml 1ug As.
- B-8 **PREPARATION OF CALIBRATION CURVE (0-10 ug):**
- B-8.0 Set apparatus as in attached diagram on page 3
- B-8.1 Place lead acetate impregnated wool in the neck of arsine generation flask.
- B-8.2 Add 50ml water in Arsine generation flask. Add 2.5ml of 1ppm Arsenic solution. This is equivalent to 2.5ug As.
- B-8.3 Add 5ml conc. HCl. Mix well.
- B-8.4 Add 2ml KI solution (2.1).
- B-8.5 Add 8ml SnCl₂ solution (2.) swirl the flask and allow standing for 15 minutes.
- B-8.6 Take 25ml of silver diethyl thiocarbamate solution in arsenic absorber.
- B-8.7 Add 5g zinc granule in Arsenic generation flask and immediately insert the cover of flask.
- B-8.8 Slowly pass N₂ through this flask @ about one bubble in 1-2 second.
- B-8.9 Allow the N₂ to pass for 45 minutes.
- B-8.10 Allow the N₂ to pass for 45 minutes.
- B-8.11 Transfer the absorber solution to 25ml vol. flask and make up to the mark with silver diethyl dithiocarbamate solution.
- B-8.12 Measure absorbance at 520nm against silver diethyl dithiocarbamate solution as blank using 50mm cell.
- B-8.13 Similarly repeat above procedure (3.4-3.11) by taking 5 ml, 7.5ml and 10ml of 1ppm As standard solution. These are equivalent to 5, 7.5 and 10 ug As.

- B-8.14 Plot calibration curve of absorbance against ug of As and calculate slope.
- B-9 **METHOD**
- B-9.1 Dissolve about 1 to 2 g fertilizer sample in demin/distil water and make up to 1L in volumetric flask.
- B-9.2 Take appropriate quantity of sample containing As not more than 10ug or use dilute sample.
- B-9.3 Proceed through 3.1-3.2 & 3.4-3.11.
- B-10 **CALCULATION**
- As (ug) = Abs. × slope × dilution factor

Calculate ppm or percentage from micrograms (ug) of As.



Alternate Method

DETERMINATION OF ARSENIC BY INDUCTIVELY COUOKED PLASMA OPTICAL EMISSION APECTROSCOPY (ICP-OES)

SAMPLE PREPARATION

Solid samples for ICP spectroscopy are first dissolved in appropriate acid by digestion process or dry.

DRY ASHING:

APPARATUS

Grinder
 50ml volumetric flask
 Fused silica crucible with lid (Boron free)
 Filter paper Whatman No. 42
 Plastic bottles
 Funnels with stand
 Balance

PROCEDURE

Weight 1.0 g ground material into boron free fused silica crucible. Place crucible in muffle furnace and increase the temperature gradually upto 450-500 °C for at least 2 hours. Cool the furnace. Remove the fused samples and add 10 ml of 0.7 N H₂SO₄. Mix well and allow to stand for 1 hour. Filter the sample in 50 ml volumetric flask using whatman No. 42. Give two to three washing to the crucible with 5.0 ml 0.7 N H₂SO₄. Make the volume upto mark with distilled/deionised water.

WET ASHING: DOUBLE ACID METHOD (HClO₄ AND HNO₃)**REAGENTS**

Perchloric acid and Nitric Acid mixture (1:2): Add 500 ml (70%) perchloric acid in 1000 ml HNO₃ (69-71 %), mix well, cool and store in an amber glass bottle.

PROCEDURE

This digestion method is used for multi-element analysis. This method can be used preferably for flame emission spectrophotometer. Weigh 1.0 g or less material in conical flask (50 ml capacity) add 3.5 ml of double acid mixture. Shift the flask on hot plate. Brown fumes of nitrate will come out. With the passage of time formation of brown fumes will decrease and the colour of the content in the flask will become light yellow. Continue heating at 175 °C until clear transparent solution formed with white dense fumes at the end. These copious white fumes will be the indication of complete digestion process. Cool the conical flask. However, this clear solution may have slight amber to yellowish tint which usually disappears when distilled water is added. By using the volumetric flask make the volume 100 ml with distilled water or as desired when contents in the conical flask is still warm/taped. Filter if necessary. Transfer to the plastic bottles, store in a cool and dry place. The digests are ready for analysis.

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References:

PS: 36-1975	Specification of Ammonium Sulphate (Fertilizer Grade)
IS: 826-1980	Specification of Ammonium Sulphate (Fertilizer Grade)
SLS: 620-1987	Specification of Ammonium Sulphate (Fertilizer Grade)