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PAKISTAN STANDARD SPECIFICATION

FOR

DIAMMINOUM PHOSPHATE



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Karachi.

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FOR
DIAMMONIUM PHOSPHATE

FOREWORD

- 0.1 This Pakistan Standard was adopted by the Pakistan Standards Institution on 30-03-1994 after the draft finalized by the Fertilizer and Allied Products Technical Committee had been approved by the Chemical National Standards Committee.
- 0.2 While preparing this standard the view of the producers, consumers and technologists have been taken into consideration and also is the existing trade practices in this field in the country.
- 0.3 For the purpose of deciding whether the requirements 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 "METHOD OF ROUNDING OFF NUMERICAL VALUES". The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.
- 0.4 This standard is intended mainly to cover the technical provisions relating to supply of material and it does not cover all the necessary provisions of a contract.

In order to keep abreast with the progress of trade and Industry Pakistan Standards are revised periodically. Suggestions from the members are welcomed and will be placed before the committee for consideration at the time of revision.

1. SCOPE

- 1.1 This Pakistan Standard prescribes the requirements and methods of test for Diammonium Phosphate for use as fertilizer.

2. SAMPLING:

- 2.1 Representative samples of the material shall be drawn as prescribed in Appendix "A" of PS: 933 – 1973.

3. REQUIREMENTS

- 3.1 The Diammonium phosphate shall also comply with the requirements specified in the Table when tested according to the methods prescribe in Appendix "B". Reference to relevant clauses of Appendix B is given in Col. 4 of the Table.

TABLE OF REQUIREMENTS:

S.#	CHARACTERISTICS	REQUIREMENTS	METHODS OF TEST & APPENDICES
1	Physical condition	Granular, free flowing Visual	
2	Moisture content % by wt. (Max)	1.5	B ₁ & B ₂
3	Nitrogen content % by wt. (Min)	18	B ₃
4	Total Phosphate (as P ₂ O ₅) % by wt. (Min). (Content of min of which 90% water soluble) soluble.)	46	B ₄
5	Screen size (Tyler)	85 % minus 6 plus 16 mesh, minimum (85% between 1 – 3 mm)	

4. PACKAGING & MARKING

- 4.1 **PACKING:** - The material shall be packed in natural/synthetic fiber bags lines with multi-walled paper or suitable plastic material or in such other suitable containers as agreed to between the purchase and the vendor. The weight of the material in a bag should ordinarily be 50 kg.
- 4.2 **MARKING:** - The container shall be securely closed and marked with the following information:-
- Name of the material.
 - Minimum Nitrogen and phosphate content of the material.
 - Name of the manufacturer
 - Weight of the material in the containers.
 - Recognized trade mark, if any, and
 - Water solubility.

APPENDIX - B**B. 1 DETERMINATION OF MOISTURE (OVEN METHOD)****B-1.1 Apparatus**

B.1.1.1 **Weighing bottle:-** Size 50 mm × 30 mm, fitted ground glass stopper with a hole.

B.1.1.2 **Air oven:-** heated electrically with temperature control system.

B.1.1.3 **Mortar:-** made of porcelain or glass, size 102 mm (4 inch) into diameter.

B-1.2 Procedure:-

B-1.2.1 Preparation of Sample:- Place about 225 gram of sample in a mortar & grind quickly. Particle size required is less than 1 weigh 5 gram of the ground sample into a weighing bottle using an analytical balance.

B-1.2.2 Determination:- Place the weighing bottle containing be sample in the air oven maintained at $100 \pm 2^{\circ}\text{C}$. After 4 hours take the sample bottle out, and cool in a desiccators for 15-20 minutes. Silica-gel is desirable as desiccating agent. Reweiat the sample using analytical balance.

B.1.3 Calculations

$$\text{Moisture percentage} = \frac{(A - B)}{A} \times 100$$

Where,

A = Weight in gram before heating

B = Weight after heating.

ALTERNATE METHOD**B.2 DETERMINATION OF WATER IN DIAMMONIUM PHOSPHATE FERTILIZER****B.2.0 SCOPE**

Titrimetric determination of 0.3-30 mg water by Karl Fischer Reagent.

B.2.1 PRINCIPLE

Water is extracted from the sample and titrated with Karl Fischer Reagent.

B.2.2 REAGENTS

1. Karl Fischer Reagent (Stabilized Grade) water equivalent approx. 3 mg H₂O / ml. Keep the reagent in a brown bottle. Water equivalent must be determined at least once / day.
2. Methanol, water content 0.02 % (m/m).

B.2.3 SAMPLE

Analysis is performed on the sample, as received; usually 10 grms sample is used for determination.

B.2.4 APPARATUS

1. Potentiometer Titrator; equipped with magnetic stirrer auto control.
2. Automatic pipette, 100 ml.
3. Magnetic Stirrer.

B.2.5 STANDARDIZATION OF KARL FISCHER REAGENT

Karl Fischer Reagent is standardized with sodium tartarate.

Transfer 20 ml of methanol to the dry titration vessel.

Neutralize methanol with Karl Fischer reagent, to end point, per instructions of manufacturer of apparatus.

Introduce 160 mg of sodium tartarate ($\text{Na}_2 \text{C}_4 \text{H}_4 \text{O}_6 \cdot 2\text{H}_2\text{O}$) into the titration vessel, containing neutralized methanol.

Titrate against Karl Fischer reagent to end point (consumption) V_0 ml).

B.2.6 Calculations of Water Equivalent (T)

Calculate water equivalent as under:

$$T = \frac{15.66 \times a}{100 \times V_0}$$

Where,

T = water in mg, equivalent to 1 ml of Karl Fischer Reagent.

a = amount of Sodium Tartarate dehydrate in mg.

V_0 = ml of Karl Fisher solution, consumed in standardization.

B.2.7 PROCEDURE

Dry 150 ml conical flask with a ground in stopper by heating at 120°C.

Allow the flask to cool in a desiccators. Transfer 10 g of sample to the flask. Add 100 ml of methanol by means of automatic burette. Place a dry soft iron stirring rod in the flask and immediately stopper the flask. Start the stirrer moter and stir until the sample has completely disintegrated (using about ½ hours stirring is required). Allow the non-dissolved material to settle. (Usually ½ hr is sufficient). Transfer of clear sample methanol solution to the dried titration vessel. Titrate against Karl Fischer reagent to the end point, following the

instructions of the manufacturer of the apparatus. (Contention V_2 ml.). Also run a blank with 20 ml of methanol (Contention V_1 ml).

B.2.8 CALCULATION:

Calculate water content of sample as under:

$$\text{Moisture \%} = \frac{(V_2 - V_1) \times T}{20}$$

Where,

V_1 = ml, Karl Fischer Reagent consumed in blank run.

V_2 = ml, Karl Fischer Reagent consumed in sample run.

T = Water in ml; equivalent to 1 ml of Karl Fischer Reagent.

B.3 DETERMINATION OF NITROGEN IN DIAMONIUM PHOSPHATE

B.3.0 SCOPE

Determination of Nitrogen at most 200 mg of Nitrogen.

B.3.1 PRINCIPLE

Ammonia is released with Sodium Hydroxide, distil and collect in a known amount of Sulphuric acid. Excess of acid is titrated with sodium hydroxide solution. The acid consumption is a measure of amount of nitrogen content.

B.3.2 REAGENTS

1. Sulphuric Acid 0.1 N, to which 60 ml of Methyl Red – Methylene blue, as indicator have been added per 10 liter.
2. Sodium Hydroxide solution 0.1 N.
3. Sodium Hydroxide Solution 10 N.
4. Methyl Red-Methylene Blue Indicator. Dissolve 1.30 g of Methy Red in one liter of 90% Ethanol. Dissolve 0.825 g of Methylene blue in one liter of 90% Ethanol. Mix the two solutions in equal volumes.

B.3.3 APPARATUS

Distillation apparatus for determining total nitrogen and Ammonical Nitrogen. See Fig. Distillation apparatus shall consist of Alkali-resistant, glass Rubber stoppers must be

renewed periodically and should fit closely in the neck of the distillation flask, to prevent condensation of liquid between glass wall and the stopper..

B.3.4 **SAMPLE**

Grind the product to minus 1 mm. Use 5 g of sample.

B.4.5 **PROCEDURE**

Total Nitrogen

Transfer 5 g of sample to a 500 ml measuring flask. Add about 300 ml of distilled water, shake contents of flaks for a few minutes, make up to mark, with distilled water, mix and filter. Pipette 50 ml of filtrate into distillation flask and dilute with distilled water to 300 ml and add a few boiling chips.

Transfer 100 ml of Sulphuric acid 0.1 N to the receiver through automatic pipette / burette and content receiver to the apparatus. Transfer 30 ml of sodium hydroxide 10 N, to the distillation flask (When adding sodium hydroxide be taken to prevent contact of sodium hydroxide with part of the neck of flask A, into h which the rubber stopper is to be inserted).

Connect the flask immediately to the apparatus Heat contents of flask at such a rate that contents of flask start boiling after 20 minutes. Distill about 200 ml. Remove the receiver from the apparatus and wash the contents of siphon and adopter into the receiver. (Immediately after distillation, check by means of an indicator paper, whether the subsequent distilled is free of ammonia).

Also run a blank, (consumption V_0 ml).

B.3.6 **Calculation.**

Calculate the amount of Total Nitrogen in % using the formula:

$$\text{Nitrogen \%} = (V_0 - V) \times 0.28$$

V_0 = ml of sodium hydroxide solution 0.1N, Consumed in blank.

V = ml of sodium hydroxide solution 0.1N, Consumed in determination.

B.4 **DETERMINATION OF TOTAL P₂O₅ IN DIAMMONIUM PHOSPHATE FERTILIZER**

B-4.0 **PRNCIPLE**

In acid medium ortho phosphate forms a yellow coloured comples of Ammonium Phospho-Vanado-molybdate with Ammonium Vanado-molybdate solution and colour intensity is matched with that of standard phosphate solution at 422 nm.

B-4.1 REAGENTS**B-4.1.1 Ammonium Vanado Molybdate Solution**

Dissolve 1 gm of Amm. Metavandadate & 20 gms of pulverized Amm. hepta molybdate in 300 ml of distilled / demin water at 50°C, in separate 500 ml beaker.

Transfer 140 ml of HNO₃ (1.37) to one litre measuring flask, add fistly Amm. Metavandadate solution with shaking & then add Amm. Molybdate solution and mix.

Make up to mark, with distilled / demin water water and mix. Store in brown bottle.

B-4.1.2 Nitric Acid-Hydrochloric Acid Mixture

Mix 8 parts distilled / demin water, 4 parts HNO₃ (1.20) and 3 parts HCl (1.25) (on volume basis).

B-4.1.3 Standard P₂O₅ Solution

Transfer 38.348 gm of KH₂ PO₄ (dried at 105°C for three hrs) to one liter measuring flask and add 500 ml distilled water then 25 ml of 10% H₂SO₄ and mix. Make up to the mark and mix
1 ml = 20 mg P₂O₅.

Transfer 10 & 15 ml of above solution thro. Burette into separate 500 ml measuring flask. Make up to mark with distilled demin water and mix. Respective solutions would contain 4 mg, 6 m P₂O₅/ 10 ml of solution marked as A & B.

B-4.2 SAMPLE

Grind the fertilizer to minus 1 mm, use 1 gm of sample in the determination.

B-4.3 PROCEDURE

Weigh 1 gm of sample and transfer to 250 ml breaker. Add 10 – 20 ml distilled / demin water and 25 ml of Niric Acid-Hydrochloric Acid mixture. Boil for 7 minutes.

Cool and transfer the contents to 1000 ml measuring flask. Rinse the beaker with water, adding rinsing to the measuring flask. Now make up to the mark and mix.

Filter through filter paper, reject first 50-60 ml of filtrate and collect the filtrate in a beaker.

Transfer 10 ml of filtrate to 100 ml measuring flask and add about 50 ml of distilled/demin water and mix.

Transfer 10 ml of standard P₂O₅ solution A & B to separate 100 ml measuring flasks and add to each about 50 ml of distilled demin water.

Add to each measuring flask, 25 ml Amm. Vanadomolybdate solution mix and make up to the mark with distilled / demin water.

Mix well and allow to 10 minutes for colour development.

Set spectrophotometer absorbance 0.000 at 422 nm with colour development with 4 mg P₂O₅ standard solution.

Measure absorbance of colour developed with 6 mg P₂O₅ standard solution and sample solution (Absorbance must be measure within 45 minutes as afterwards colour fading takes place).

Measure absorbance of colour developed with sample solution.

B-4.4 CALCULATIONS

Calculate Total P₂O₅ contents as under:

$$\text{Total P}_2\text{O}_5 : \% = 4 + \left(\frac{S}{B} \times 2 \right) \times 10$$

Where,

B = Absorbance of Standard P₂O₅ Solution B.

S = Absorbance of sample solution.

B-5 DETERMINATION OF WATER SOLUBLE P₂O₅ IN DIAMMONIUM PHOSPHATE FERTILIZER

B-5.1 PRINCIPLE

Fertilizer sample is shaken with water and filtered. Aliquot of filtrate is boiled with nitric acid & phosphate content is determined, by forming yellow colored complex of Ammonium-phospho-vanado molybdate with Amm. Vanado molybdate solution and color intensity is measured with that of standard phosphate solution at 422 nm.

B-5.2 REAGENTS

1. Ammonium Vanado Molybdate Solution

Dissolve 1 gm of Amm. Metavandate & 20 gms of purified Amm hepta molybdate in 300 ml of distilled water at 50°C, in separate 50 ml beaker.

Transfer 140 ml of HNO₃ (1.37) to one litre measuring flask, add firstly Amm. Metavandate solution with shaking & then and Amm. Molybdate solution & mix.

Make up to mark with distilled water & mix. Store in brown bottle.

2. Nitric Acid 1 M

3. Standard P₂O₅ Solution

Transfer 38.348 gms of KH₂PO₄ (dries at 105°C for three hrs) to one litre measuring flask & add 500 ml distilled water, then 25 ml of 10% P₂O₅ and mix.

Transfer 5 & 10 ml of above solution thru, burette into separate 500 ml measuring flasks. Make up to mark with distilled water and mix. Respective solution would contain 2 mg & 4 mg P₂O₅ / 10 ml of solution marked as A & B.

SAMPLE

Grind the sample to minus 1 mm, use 1 gms of sample in the determination.

B-5.3 PROCEDURE

1. Transfer 1 gms of sample to a 1000 ml measuring flask. Add about 900ml of water and shake for ½ hour at 30-40 revolutions per minutes.
2. Make up to mark, mix and filter: reject first 50-60 ml of filtrate & collect the remainder in a beaker.
3. Transfer 5 ml of filtrate into a 250 ml Tall form beaker add 25 ml nitric acid 1 M and boil gently for 5 minutes.
4. Cool & transfer contents of beaker & washing into a 100 ml measuring flask.
5. Transfer 10 ml of standard P₂O₅ Solution A & B to separate 100 ml measuring flask & add to each about 50 ml of distilled water.
6. Add to each measuring flask, 25 ml Amm. Vanadab molybdate solution mix and make up to the mark with distilled water.
7. Mix well & allow to stand for 10 minutes for color development.
8. Set spectrophotometer absorbance 0.000 at 422 nm, with color developed with 2 mg P₂O₅ standard solution.
9. Measure absorbance of color developed with 4 mg P₂O₅ standard solution.
Absorbance must be measured within 45 minutes as afterwards color fading takes place.
10. Measure absorbance of color developed with sample solution.

B-5.6 CALCULATION

Calculate water soluble P₂O₅ as under:-

$$\% \text{ Water Soluble P}_2\text{O}_5 = 2 + \left(\frac{S}{B} \times 2 \right) \times 20$$

Where,

B = Absorbance of standard P₂O₅ Solution B.

S = Absorbance of sample solution.

NOTE

In case shaking machine is not available, sample solution may be prepared, following the under mentioned procedure.

- Place 0.5 gms of sample on a 11-CM citer and wash with successive small portions of water, collecting the filtrate in a 500 measuring flask, continue washing till filtrate measure 200-225 ml. Allow each portion of wash water to pass thru the filter, before adding more and use suction if, after 30 minutes it is obvious that washing cannot be completed in 1 hour.
- If the filtrate is turbid, add 1- 2 ml of nitric acid, dilute to mark and mix well.
- Proceed from steps 3 onwards.