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PAKISTAN STANDARD SPECIFICATION

FOR

TRIPLE SUPER PHOSPHATE (2nd Revision)



**PAKISTAN STANDARDS AND QUALITY CONTROL AUTHORITY,
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Plot No. ST-7A, Block-3, Scheme 36, Gulistan-e- Johar
Karachi.**

**PAKISTAN STANDARD SPECIFICATIONS
FOR
TRIPLE SUPER PHOSPHATE (2nd Revision)**

0. FOREWORD

- 0.1 This Revised Pakistan Standard was adopted by the Pakistan Standards and Quality Control Authority on 11th February, 2009 approval by the Chemical National Standard Committee and the draft finalized by the Fertilizer & Allied Products Technical Committee
- 0.2 While preparing this standard the Technical Committee responsible for the preparation of this standard gave due consideration to the views of the producers, consumers and technologists and felt that it should be related to the prevailing trade and manufacturing practices followed in this field in the country.
- 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 PS: 103: 1991 "method of Rounding Off Numerical Value"; the number of places retained in the rounded off value should be the same as those of the specified value in the standard.
- 0.4 This standard is intended chiefly to cover the technical provisions relating to the supply of the material, and it does not include all the necessary provision of a contract.
- 0.5 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 committees for consideration at the time of revision.

1. SCOPE

- 1.1 This standard prescribes the requirements and the methods of sampling and test for triple super phosphate.

2 SAMPLING

- 2.1 Representative test samples of the material shall be drawn and prepared as prescribed in Appendix "A"

3. REQUIREMENTS:

- 3.1 Description - The material shall be granular, free from excessive lumps, shall not form hard cakes on storage and also free from visible matter.
- 3.2 The material shall comply with the requirements given in Table-I. When tested according to the methods prescribed in Appendix "B". References to the relevant clauses of Appendix "B" are given in Col. 4 of the Table 1

TABLE I

Requirements for Triple Super Phosphate

S.#	Characteristics	Requirements	Method of test Ref. to Cl. # of Appendix "B"
a	B	C	d
1.	Physical condition	free flowing granular	Visual inspection
2.	Moisture, percent by weight, (max).	4.0	B-3
3.	Free phosphoric acid (as P ₂ O ₅) percent By weight. (max)	3.0	B-4
4.	Water soluble phosphate (as P ₂ O ₅) percent by weight, (min)	43.0	B-5
5.	Phosphate content available (as P ₂ O ₅) percent by weight,(min).	46.0	B-6
6.	Size distribution	80% between 1 & 3 mm & 97% between 1 & 4 mm	B-7

4. PACKING AND MARKING**4.1 Packing**

4.1.1 The material shall be packed and supplied in sound, strong, moisture proof packages or container (natural/synthetic fiber bags are of multi wall paper with bitumen or polyethylene moisture - proofing layer. Mono film bags of heavy (0.15-0.2 mm thickness) polyethylene also are satisfactory or in such other suitable containers) as agreed to between the purchaser and the vendor. Jute or woven polypropylene bags with monofilm plastic liners shall also be used.

4.1.2 The weight of the material in a bag should ordinarily be 50 kg net.

4.1.2.3 Marking

4.1.2.4 The packages shall be securely closed and marked with the following information:-

- a) Name of the material.(Triple Super Phosphate Fertilizer Grade)
- b) % age of available phosphate (P₂O₅) and water soluble phosphate (P₂O₅) content of the material.
- c) Name and address of the manufacturer & recognized trade mark if any.
- d) Net Weight in Kg of the material in the container.
- e) Best before use.

APPENDIX "A".**SAMPLING OF TRIPLE SUPER PHOSPHATE (FERTILIZER)****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 Sample shall be taken at a place protected from damp air, dust and soot.
- A.1.2 The sampling apparatus shall be clean and dry when used.
- A.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.
- 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 air-tight 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 air-tight after filling and marked with full details of sampling, the date of sampling, year of manufacture, and other important particulars of the consignments.

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 Clause 1 and 2 of Table. II.

TABLE II**NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING**

Lot Size N (1)	No. of containers to be selected n (2)
Up to 100	5
101 to 300	6
301 to 500	7
501 to 800	8
801 to 1300	9
1301 to 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.2.3.1 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 r th container thus counted shall be with drawn and all such containers shall constitute the sample.

A.3 **TEST SAMPLE AND REFEREE SAMPLE**

A.3.1 Draw with an appropriate sampling apparatus small portions of the material from different parts of the containers selected, the total quantity taken out from each container shall be sufficient to conduct the tests for characteristics given in 3.

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

A.3.3 All individual test sample and composite test sample, shall be divided into three equal parts, thus forming three sets of test sample. These parts shall be immediately transferred to appropriate air tight container. These shall be labeled with all the particulars of sampling giving under A. 1.7. One of these sets of test samples shall be sent to the purchaser and another to the vendor.

A.3.4 Referee Samples. — The third set of test samples, bearing the seals of the purchaser and the vendor, shall constitute the referee samples 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 TESTS**

A.4.1 Test for the determination of soluble phosphates shall be conducted on each of the individual test samples.

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

A-5 **CRITERION FOR CONFORMITY**

A-5.1 The test results for total water soluble phosphates shall be recorded as shown in table III. The mean the range of the test results shall be calculated as follows:-

$$\text{Mean } (x) = \frac{\text{The sum of the test result's}}{\text{Number of test results}}$$

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 this characteristic. If the condition given Col. 6 of Table III is satisfied, the lot shall be declared to have satisfied the requirement for the characteristic.

A-5.2 A lot shall be declared as conforming to the specification only when it has satisfied each of the requirements specified in 3.

TABLE III**CRITERION FOR CONFORMITY**

Sr.#	Characteristic	Test Results 1.2...n	Mean	Range	Criterion for Conformity
1	2	3	4	5	6
	Water soluble phosphates (as P ₂ O ₅) % by weight	----	X	R	$X - 0.6 R \geq$ the value specified in Table-1 (3)
	Available Phosphate (as P ₂ O ₅) % by weight	----	do	do	do

APPENDIX "B"**ANALYSIS OF TRIPLE SUPER PHOSPHATE****B.1 QUALITY OF REAGENTS**

B.1.1 Unless specified otherwise, pure chemicals and Water (see 593:1991 "Specification for water, Distilled Quality (Revised) shall be used in tests.

NOTE: - Pure chemicals shall mean chemical that do not contain impurities which affect the results of analysis.

B.2 PREPERATION OF SAMPLE

B.2.1 Place composite sample in an air-tight container and deliver entire sample to laboratory. Composite sample is reduced by using riffle divider to an amount sufficient for analysis. Sieve through PS: Sieve 18 aperture 1 mm (see Pakistan Standard Specification for test

sieve PS 392: 1964) breaking the lumps. Grind in a mortar the part remaining on the sieve until all particles pass through. Mix thoroughly, transfer immediately to a wide-mouth bottle and preserve this prepared sample for analysis.

B.2.1.1 The grinding and-sieving shall be done as rapidly as possible to avoid gain or loss of moisture.

B.3 DETERMINATION OF MOISTURE**B.3.1 Principle**

The moisture content of sample can be determined by drying the sample in an oven at 105^oC till constant weight is obtained. The loss in weight after drying is moisture.

B.3.2 Equipment and Glass ware

1. Electric Oven.
2. Weighing bottle.
3. Desiccators.
4. Analytical Balance.

B.3.3 Procedure

- B.3.3.1 The fertilizer samples placed in oven at 105°C for 4— 8 hours or till constant weight to drive off all fertilizer moisture. Sample is weighed before and after drying and moisture content is calculated on oven dry basis.
- B.3.3.2 Weigh accurately 4— 5 g of sample in clean oven dried weighing bottle. Remove the lid and place in the oven at 105°C for 4 — 8 hours or until constant weight is obtained.
- B.3.3.3 Then remove the weighing bottle covered with the lid and place it in desiccators for 30 minutes in order to cool it. After cooling weigh the bottle . Percent moisture may be calculated as follows:

Calculation:

$$\% \text{ Moisture} = \frac{\text{Wet weight} - \text{dry weight} \times 100}{\text{Wet weight}}$$

B.4 DETERMINATION OF FREE PHOSPHORIC ACID

- B.4.1.1 Acetone — Specification for Acetone.
- B.4. 1.2 Standard Sodium Hydroxide Solution — 0.1 N.
- B.4.1.3 Bromoceresol Green Indicator Solution:- Dissolve 0.1 g of bromoceresol green in 100 ml of rectified spirit.
- B.4.2 Procedure - Weigh accurately about 2.5 g of the prepared sample and transfer to a soxhlet extractor. Add about 100 ml of acetone and extract for three hours. Cool and distill of the acetone as far as possible. Take up the residue with water and make up the volume to 250 ml. Pipette out exactly 100 ml of this solution and titrate with standard sodium hydroxide solution,(0.1N) using bromoceresol green as indicator, until the colour just changes from yellow to blue.

B.4.3 Calculation.

$$\text{Free phosphoric acid (P}_2\text{O}_5\text{), by \% by weight; } = \frac{V \times N \times 35.5 \times 250 \times 100}{W \times 100 \times 1000}$$

where

V = volume in ml of standard sodium hydroxide solution used.

N = normality of standard sodium hydroxide solution, and

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

B.5 Determination of water soluble P₂O₅ in Triple Super Phosphate by Spectrophotometer

- B -5.1 Apparatus/Equipments:
- B.5.1.1 Spectrophotometer with 10 mm cell
- B.5.1.2 Volumetric flasks : 100 ml, 1L
- B.5.1.3 Beaker : 400 ml

B.5.1.4	Pipettes	:	10 ml, 20 ml
B.5.1.5	Conical Flask	:	250 ml
B.5.1.6	Watch Glass		
B.5.1.7	Funnel		

B.5.2. CHEMICALS/REAGENTS:

B.5.2.1 Nitric Acid conc. (Sp. Gr = 1.38)

B.5.2.2 Molybdovanadate Reagent
In a 1000ml volumetric flask dissolve 175 ml HNO₃ (conc) in 500 ml demin/distill water make the volume upto mark homogenize.

B.5.2.3. Dissolve 2.5 g Ammonium metavanadate (NH₄ VO₃) in 500 ml boiling water. Add 20 ml of HNO₃ (Conc) and after cooling dilute to 1000 ml.

B.5.2.4 Dissolve 50 g Ammonium Molybdate (NH₄)₆ MO₇ O₂₄ 4H₂O in 750 ml of hot demin/distill water. After cooling dilute to 1000 ml with demin/distill water.

B.5.2.5 Mix equal volumes of these solutions in order 2.2.1, 2.2.2, 2.2.3 at room temperature and store in amber bottle.

B.5.3 P₂O₅ Standard Solution (1000 ppm)

B.5.3.1 Dissolve 1.9155 g Potassium Dihydrogen Phosphate previously dried for two hours at 110 °C in 1L volumetric flask containing demin/distill water, mix & make upto mark.

B.5.4 Preparation of Calibration curve (0-5 mg)

B.5.4.1 Take a series of 100 ml volumetric flasks already containing 25 ml of molybdovanadate reagent and 1.3 ml of conc. HNO₃

B.5.4.2 Take an aliquote of 1,2,3,4 & 5 ml of P₂O₅ standard solution in these flasks.

B.5.4.3 Make the volume upto mark with demin water mix & wait for 15 minutes.

B.5.4.4 Make reagent blank with 25 ml Molybdovanadate reagent and 1.3 ml conc. HNO₃ & making volume upto mark with demin water.

B.5.4.5 Measure the absorbance at 430 nm against reagent blank with 10 mm cell.

B.5.4.6 Draw the calibration curve between Abs, & mg P₂O₅ and find the slope.

B.5.5 PROCEDURE/METHOD

B.5.5.1 Weigh about 2.5 g of TSP sample grinded through 0.5 mm mesh in 400 ml beaker.

B.5.5.2 Dissolve the grinded sample in a volumetric flask containing demin/distill water and make the volume upto mark.

B.5.5.3 Put magnetic stirrer in the flask and keep the stirring for 15 minutes.

B.5.5.4 Filter the above sample in 250 ml conical flask.

B.5.5.5 Dilute 20 ml of filtered solution upto 100 ml with demin water.

B.5.5.6 Take a 100 ml volumetric flask containing 25 ml of molybdovanadate reagent and 1.3 ml conc.

HNO₃

B.5.5.7 Add 10 ml solution from 4.5

B.5.5.8 Proceed thru 3.3 and 3.4

B.5.6 **CALCULATION**

$$P_2O_5 \% = \frac{\text{Abs x slope x 1000 x 100 x 100}}{\text{Wt of sample x 20 x 10 x 1000}}$$

OR

$$P_2O_5 \% = \frac{\text{Abs x slope x 50}}{\text{Wt of sample}}$$

ALTERNATE METHOD**B.6** **DETERMINATION OF WATER SOLUBLE P₂O₅ IN TSP BY TITRATION METHOD.****B.6.1** **APPARATUS/ EQUIPMENTS:**

B.6.1.1	Conical flask	:	250 ml
B.6.1.2	Volumetric flasks	:	1L
B.6.1.3	Beaker	:	250ml
B.6.1.4	Pipette	:	50 ml
B.6.1.5	Cylinder	:	25 ml

B.6.2 **CHEMICAL/ REAGENTS:****B.6.2.1** **Sodium Hydroxide Solution (1.0N)**

Dissolve 40 g of NaOH pellets in 1000 ml volumetric Flasks and make the volume up to the mark, Standardize with 1.0 N Potassium Hydrogen Phthalate using phenolphthalein indicator.

B.6.2.2 **Sodium Hydroxide Solution (0.1 N)**

Dissolve 4.0g of NaOH pellets in 1000 ml volumetric Flasks and make the volume up to the mark, Standardize with 0.1 N Potassium Hydrogen Phthalate using phenolphthalein indicator.

B.6.2.3 **Bromocresol Green Indicator (0.1%)**

Dissolve 0.1 g of solid in 100ml ethanol.

B.6.2.4 **Tashiro Indicator**

Dissolve 0.1g of methyl red in 100ml ethanol.
Dissolve 4ml of 1% methylene blue solution in 96ml of ethanol.
Mix (a) & (b).

B.6.2.5 Lanthanum Nitrate (4.0%)

Dissolve 40g of La (NO₃)₃. 6H₂O in 1000 ml volumetric flask with demineralized water and make upto mark.

B.6.2.6 METHOD / PROCEDURE

- a). Take 2.5g sample grinded through 0.5mm mesh in 250ml beaker. Add water & mix to dissolve.
- b). Transfer quantitatively into 1L volumetric Flask, make the volume up to mark & mix thoroughly.
- c). Put a magnetic stirrer in the flask and keep the stirring for 15 minutes.
- d). Filter the above solution and take 50ml with help of pipette in a 250ml conical flask.
- e). Add three drops of Bromocresol green indicator.
- f). Add 1 .0ml of 1 .0N NaOH solution and the 0.1N NaOH till greenish blue color is obtained.
- g). Add 25 ml of La (NO₃)₃. 6H₂O solution.
- h). Add 10 drops of tashiro indicator.
- i). Titrate with 0.1N NaOH till appearance of greenish color.
- j). Note milli litres of 0.1 N NaOH used as "V"ml.

B.6.2.7 CALCULATION:

$$P_2O_5 \% = \frac{Vml \times 0.1 \times 35.5 \times 1000 \times 100}{Wt. \text{ of sample} \times 50 \times 1000}$$

OR

$$P_2O_5 \% = \frac{V \text{ ml} \times 7.1}{Wt. \text{ of sample}}$$

B.7 DETERMINATION OF CITRATE INSOLUBLE P₂O₅ & AVAILABLE P₂O₅ IN TRIPLE SUPER PHOSPHATE PRODUCTS BY SPECTROPHOTOMETER**B.7.0 APPARATUS/ EQUIPMENTS:**

- i. Spectrophotometer with 10mm cell
- ii. Volumetric flasks : 100ml,1L
- iii. Beaker : 400ml
- iv. Pipettes : 10ml, 20ml
- v. Conical Flask : 250 ml
- vi. Watch Glass
- vii. Funnel
- viii. Filter Paper whatman No. 2 or whatman 40.
- ix. Filter Paper whatman No. 5 or whatman 42 or 44.

B.7.1 CHEMICALS / REAGENTS:**B.7.1.1 Perchloric acid**

B.7.1.2 Molybdovanadate Reagent

B.7.1.2.1 In a 1000ml volumetric flask dissolve 1gram ammonium metavanadate ($\text{NH}_4 \text{VO}_3$) in 125 ml hot water, cool, add 125 ml perchloric acid and cool again.

B.7.1.2.2 In another vessel dissolve 20g Ammonium molybdate tetrahydrate in demineralize water. Slowly add this solution, with stirring to the metavanadate solution and add 100 ml more HClO_4 . Allow standing overnight and dilute to 20 liters.

B.7.1.3 P_2O_5 Standard Solution (1000 ppm)

B.7.1.3.1 Dissolve 1.9155g Potassium Dihydrogen Phosphate previously dried for two hours at 110°C in 1L volumetric flask containing demineralize water, mix & make upto mark. "1 ml = 1 mg P_2O_5 "

B.7.1.4 Digestions acid solution

B.7.1.4.1 Four parts HNO_3 1:1 one part HCL.

B.7.2 PREPARATION OF CALIBRATION CURVE (0-5mg):

- a Take a series of 100ml volumetric flasks already containing 20ml of molybdovanadate reagent.
- b Take an aliquot of 1,2,3,4 & 5ml of P_2O_5 standard solution in these flasks.
- c Make the volume upto mark with demineralized water mix & wait for 10 minutes.
- d Make reagent blank with 25 ml Molybdovanadate reagent making volume upto mark with demineralized water.
- e Measure the absorbance at 400 nm against reagent blank with 10mm cell.
- f Draw the calibration curve between Absorbance. & mg P_2O_5 and find the slope.

B.7.3 PROCEDURE FOR CITRATE INSOLUBLE P_2O_5

B.7.3.1 Transfer a 2.0 to 2.5 g sample into a 250 ml Erlenmeyer flask or a 250ml beaker.

B.7.3.2 Add 40 ml demineralized water and still continuously for 15 minutes.

B.7.3.3 Transfer contents with a minimum amount of water onto a whatman No. 2 or whatman 40 paper or equivalent.

B.7.3.4 Wash solids with a fine stream of water, using approximately 10 ml increments, around entire periphery of paper in a circular path to ensure that the water and solids are thoroughly mixed with each addition.

B.7.3.5 Allow each portion of water to pass through paper before adding next portion.

B.7.3.6 Collect 250 ml of filtrate using suction, if necessary, to complete within one hour.

B.7.3.7 After removing water soluble P_2O_5 , transfer filter and residue to a 250 ml flask containing 100 ml citrate solution previously heated to 65°C .

B.7.3.8 Stopper flask and shake vigorously until filter paper is reduced to a pulp.

B.7.3.9 Relieve pressure in flask by removing stopper momentarily.

B.7.3.10 Continuously agitate stopped flask in a constant temperature bath at 65°C for exactly one hour.

CAUTION: When using a hot air bath, extra time should be allowed to adjust the temperature inside the air bath back up to 65°C (the action of the apparatus should be such that dispersion of

the sample in the citrate solution is continually maintained and entire inner surface of flask and stopper is continually bathed with solution).

- B.7.3.11 Remove the flask from the heating bath and immediately filter the citrate solution by suction as rapidly as possible, through a Whatman No.5 Paper or Whatman No. 42, or equivalent, using a Buchner or ordinary funnel of Shimer tubes with cone or filter pad.
- B.7.3.12 Wash sample residue with water heated to 65°C until volume of filtrate is 350 ml, allowing time for thorough draining between washings.
- B.7.3.13 If material is one that yields a cloudy filtrate, wash with 5% NH₄OH solution heated to 65°C.
- B.7.3.14 Remove paper and residue from funnel, police as necessary, and prepare for analysis by the following methods 4.2 or 4.3.

B.7.4 Method -1

- B.7.4.1 Place paper and residue in porcelain dish and dry.
- B.7.4.2 Ignite at 600°C until all organic matter is destroyed.
- B.7.4.3 Add 10-15ml HCl and digest until all phosphate is dissolved, cool solution.
- B.7.4.4 Filter quantitatively into a 100 ml volumetric flask. Make volume upto mark with demineralized water and take suitable aliquot in another 100 ml volumetric flask.
- B.7.4.5 Add 20 ml molybdovanadate reagent, dilute to volume, mix thoroughly and let stand 10 minutes.
- B.7.4.6 Prepare reagent blank and standard P₂O₅ solution at the same samples are prepared.
- B.7.4.7 Add 50 ml water.
- B.7.4.8 Add 20 ml molybdovanadate reagent, dilute to volume, mix thoroughly and let stand ten minutes.
- B.7.4.9 Read absorbance of sample immediately with blank at wave length 400 nm.
- B.7.4.10 Note the reading and calculate results.

B.7.5 Method -2

- B.7.5.1 Place paper and residue into a 250 ml flask.
- B.7.5.2 Add 25 ml digestion acid solution and heat to boiling.
- B.7.5.3 Digest for 25 minutes or until all red fumes are expelled, cool solution.
- B.7.5.4 Filter quantitatively into a 100 ml volumetric flask. Make volume upto mark with demineralized water and take suitable aliquot in another 100 ml volumetric flask.
- B.7.5.5 Add 20 ml molybdovanadate reagent, dilute to volume, shake vigorously and let stand for 10 minutes.
- B.7.5.6 Prepare reagent blank and standard P₂O₅ solution at the same samples are prepared.

B.7.6 CALCULATION FOR:

$$\text{Citrate Insoluble P}_2\text{O}_5\% = \frac{\text{Absorbance} \times \text{slope} \times 100}{\text{Wt of sample} \times \text{aliquot from 100 ml solution} \times 10}$$

B.7.7 PROCEDURE/METHOD FOR TOTAL P₂O₅:

- B.7.7.1 Weigh about 2.5g of Triple Super Phosphate sample grinded through 0.5mm mesh in 400ml beaker.
- B.7.7.2 Add 50ml of 5% H₂SO₄ and digest for 15 minutes, covering the mouth of beaker with watch glass.
- B.7.7.3 Dissolve the digested sample in a volumetric flask containing demineralized water and make the volume upto mark.
- B.7.7.4 Filter the above sample in 250ml conical flask.
- B.7.7.5 Dilute 20ml of filtered solution upto 100 ml with demineralized water.
- B.7.7.6 Take A 100 ml volumetric flask containing 25ml of molybdovanadate reagent and 1.3 ml cone. HNO₃.
- B.7.7.7 Add 10 ml solution from 4.5.
- B.7.7.8 Make the volume upto mark with demin/distill water mix & wait for 15 minutes.
- B.7.7.9 Make reagent blank with 25ml Molybdovanadate reagent and 1.3 ml cone. HNO₃ & making volume upto mark with demineralized water.
- B.7.7.10 Measure the absorbance at 430 nm against reagent blank with 10 mm cell.

B.7.8 CALCULATION FOR TOTAL P₂O₅

$$P_2O_5 \% = \frac{\text{Abs} \times \text{slope} \times 1000 \times 100 \times 100}{\text{Wt of sample} \times 20 \times 10 \times 1000}$$

OR

$$P_2O_5 \% = \frac{\text{Abs} \times \text{slope} \times 50}{\text{Wt of sample}}$$

B.7.8.1 CALCULATION FOR AVAILABLE P₂O₅

$$\text{Available } P_2O_5 \% = \text{Total } P_2O_5 - \text{Citrate Insoluble } P_2O_5$$

B.8 DETERMINATION OF CITRATE INSOLUBLE P₂O₅ & AVAILABLE P₂O₅ IN TSP BY TITRATION**B.8.1 APPARATUS/ EQUIPMENTS:**

- | | | |
|-------|-------------------------------------------------|--------------|
| i. | Volumetric flasks | 100ml,1L |
| ii. | Beaker | 400 ml |
| iii. | Pipettes | 10 ml, 20 ml |
| iv. | Conical Flask | 250 ml |
| v. | Watch Glass | |
| vi. | Funnel | |
| vii. | Filter Paper whatman No. 2 or whatman 40. | |
| viii. | Filter Paper whatman No. 5 or whatman 42 or 44. | |

B.8.2 CHEMICALS / REAGENTS:**B.8.2.1 Sodium Hydroxide Solution (1.0N)**

Dissolve 40g of NaOH pellets in 1000ml vol. Flasks and make. Standardize with 1.0N Potassium Hydrogen Phthalate using phenolphthalein indicator.

B.8.2.2 Sodium Hydroxide Solution (0.1N)

Dissolve 4.0g of NaOH pellets in 1000ml vol. Flasks and make. Standardize with 0.1N Potassium Hydrogen Phthalate using phenolphthalein indicator.

- B.8.2.3 Bromocresol Green Indicator (0.1%)
Dissolve 0.1 g of solid in 100ml ethanol.
- B.8.2.4 Tashiro Indicator
a) Dissolve 0.1g of methyl red in 100ml ethanol.
b) Dissolve 4ml of 1% methylene blue solution in 96ml of ethanol. Mix (a) & (b).
- B.8.2.5 Lanthanum Nitrate (4.0%)
Dissolve 40g of La (NO₃)₃. 6H₂O in 1000ml volumetric flask with demin water and make upto mark.
- B.8.2.6 Digestions acid solution
three parts HNO₃:1, one part HCL.
- B.8.2.7 Citrate Solution:-
Dissolve 370 g citric acid in 1.5 L distil water and 345 ml NH₄OH (28-29% NH₃). Adjust pH to 7.

B.8.3 METHOD FOR CITRATE INSOLUBLE P₂O₅

- B.8.3.1 Transfer a 2.0 to 2.5 g sample into a 250 ml Erlenmeyer flask or a 250ml beaker.
- B.8.3.2 Add 40 ml demin/distill water and stir continuously for 15 minutes.
- B.8.3.3 Transfer contents with a minimum amount of water onto a whatman No. 2 or whatman 40 paper or equivalent.
- B.8.3.4 Wash solids with a fine stream of water, using approximately 10 ml increments, around entire periphery of paper in a circular path to ensure that the water and solids are thoroughly mixed with each addition.
- B.8.3.5 Allow each portion of water to pass through paper before adding next portion.
- B.8.3.6 Collect 250 ml of filtrate using suction, if necessary, to complete within one hour.
- B.8.3.7 After removing water soluble P₂O₅, transfer filter and residue to a 250 ml flask containing 100 ml citrate solution previously heated to 65°C.
- B.8.3.8 Stopper flask and shake vigorously until filter paper is reduced to a pulp.
- B.8.3.9 Relieve pressure in flask by removing stopper momentarily.
- B.8.3.10 Continuously agitate stoppered flask in a constant temperature bath at 65°C for exactly one hour.

CAUTION:

When using a hot air bath, extra time should be allowed to adjust the temperature inside the air bath back up to 65°C (the action of the apparatus should be such that dispersion of the sample in the citrate solution is continually maintained and entire inner surface of flask and stopper is continually bathed with solution).

- B.8.3.11 Remove the flask from the heating bath and immediately filter the citrate solution by suction as rapidly as possible, through a Whatman No.5 Paper or Whatman No. 42, or equivalent, using a Buchner or ordinary funnel of Shimer tubes with cone or filter pad.
- B.8.3.12 Wash sample residue with water heated to 65°C until volume of filtrate is 350ml, allowing time for thorough draining between washings.

- B.8.3.13 If material is one that yields a cloudy filtrate, wash with 5% NH₄OH solution heated to 65°C.
- B.8.3.14 Remove paper and residue from funnel, keep watch over as necessary, and prepare for analysis by the following method.
- B.8.3.15 Place paper and residue in porcelain dish and dry.
- B.8.3.16 Ignite at 600°C until all organic matter is destroyed.
- B.8.3.17 Place paper and residue into a 250 ml flask.
- B.8.3.18 Add 25 ml digestion acid solution and heat to boiling.
- B.8.3.19 Digest for 25 minutes or until all red fumes are expelled, cool solution.
- B.8.3.20 Filter quantitatively into a 100 ml volumetric flask. Make volume upto mark with demin water and take suitable aliquot in another 250 ml volumetric flask.
- B.8.3.21 Add three drops of Bromocresol green indicator.
- B.8.3.22 Add 1.0ml of ION NaOH solution and then 0.1N NaOH till greenish blue color is obtained.
- B.8.3.23 Add 25 ml of La (NO₃)₃. 6H₂O solution.
- B.8.3.24 Add 10 drops of tashiro indicator.
- B.8.3.25 Titrate with 0.1N NaOH till appearance of greenish color.
- B.8.3.26 Note milli liters of 0.1N NaOH used as "V"ml.

B.8.4 CALCULATION FOR CITRATE INSOLUBLE P₂O₅:

$$\text{Citrate Insoluble P}_2\text{O}_5\% = \frac{\text{Vml} \times 0.1 \times 35.5 \times 1000 \times 100}{\text{Wt. Of sample} \times \text{mls taken from step 3.21} \times 1000}$$

B.8.5 PROCEDURE/METHOD FOR TOTAL P₂O₅:

- B.8.5.1 Take 2.5g sample grinded through 0.5mm mesh in 250ml beaker.
- B.8.5.2 Add 50ml of digestion acid solution and cover beaker with watch glass.
- B.8.5.3 Boil gently for fifteen minutes.
- B.8.5.4 Cool the beaker and add about 50 ml demin water.
- B.8.5.5 Transfer into 1L vol. Flask, make upto mark & mix. thoroughly.
- B.8.5.6 Filter the above solution and take 50ml with help of pipette in a 250ml conical flask.
- B.8.5.7 Add three drops of Bromocresol green indicator.
- B.8.5.8 Add 1.0 ml of 1.0 N NaOH solution and then 0.1N NaOH till greenish blue color is obtained.
- B.8.5.9 Add 25 ml of La (NO₃)₃. 6H₂O solution.
- B.8.5.10 Add 10 drops of tashiro indicator.
- B.8.5.11 Titrate with 0.1N NaOH till appearance of greenish color.
- B.8.5.12 Note milli liters of 0.1N NaOH used as "V"ml.

B.8.6 CALCULATION FOR TOTAL P₂O₅:

$$\text{P}_2\text{O}_5 \% = \frac{\text{Vml} \times 0.1 \times 35.5 \times 1000 \times 100}{\text{Wt of sample} \times 50 \times 1000}$$

OR

$$P_2O_5 \% = \frac{Vml \times 7.1}{Wt \text{ of sample}}$$

B.8.6.1 CALCULATION FOR AVAILABLE P₂O₅:

Available P₂O₅ = Total P₂O₅ - Citrate Insoluble P₂O₅

B.8.7 DETERMINATION OF SIZE DISTRIBUTION OF TRIPLE SUPER PHOSPHATE GRANULES.**B.8.7.1 Apparatus / Equipment.**

- i. Stainless Steel sieves of required mesh size with lid and bottom pan.
- ii. Sieves shaker
- iii. Top loading balance
- iv. Riffle sampler
- v. Brush

B.8.7.2 PROCEDURE

- a) First arrange the individually tare sieves in descending order of mesh size from top to bottom.
- b) Place receiving pan on the bottom of stack.
- c) Weigh about 200 to 300 g sample taken through riffle sampler
- d) Transfer sample onto the top sieve, place lid on top of sack.
- e) Place the sieve on shaker and clamp it in position.
- f) Switch on the machine and let it shake/vibrate the sieves for 5 minutes.
- g) Switch off the machine and take off the sieves one by one.
- h) Weigh sieve & sample on top loading balance.
- i) Calculate the weight of the sample retained on each sieve.

B.8.7.3 Calculation

Calculate weight percent on each sieves by following formula.

$$Wt \% \text{ on each sieve} = \frac{\text{Weight (g) on sieve} \times 100}{\text{Total weight of sample}}$$

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