

PS: 5295-2014
ICS: 65.080

PAKISTAN STANDARD SPECIFICATION
FOR
BIO ORGANO PHOSPHATE



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**PAKISTAN STANDARDS SPECIFICATION
FOR
BIO ORGANO PHOSPHATE (BOP) FERTILIZER**

0.0 FOREWORD

0.1 This Pakistan Standard 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 Division.

0.2 Bio Organo phosphate (BOP) fertilizer product provides 20% (± 2) organic P_2O_5 25% (± 5) organic matter. The product provides multiple benefits to the farmer by improving soil and crop health. The organic P component is more efficient with low P fixation tendency in soil and prolonged availability to crop than chemical fertilizer, under soil and climatic condition. The product can positively contribute in promoting balanced fertilizer use and breaking current stagnancy in crop yields thus improving agricultural productivity.

Bio Organo Phosphate (BOP) produced by an innovation and has better use efficacy due to less probability of fixation, precipitation or insolubilization than current commercial soluble chemical/inorganic fertilizers. This will help increase P use efficiency and optimize ever increasing per hectare use of phosphorous fertilizers.

0.3 The fertilizer committee responsible for the preparation of this draft felt that it is necessary to lay down the specifications on BOP fertilizer to safeguard the interests of the farmer community and to protect them from using wrong types or grades of BOP fertilizer as well safe guard the interest of the manufactures.

0.4 While preparing this standard the views of all stake holders including manufacturers, importers, testing authorities, technologist/experts and consumers have been taken into consideration and also the existing trade practice in this field in the country, by the Technical Committee. Furthermore, due weightage had given to the need for international co-ordination among standards prevailing in different countries of the world.

0.5 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.

0.6 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 Technical Committees for consideration at the time of revision.

0.7 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.

1.0 SCOPE

1.1 This standard provides requirement and methods for Bio Organo Phosphate for use as fertilizer. The material is known as BOP.

2.0 REQUIREMENT:

2.1 The material shall consist essentially of BOP and shall be in the form of free flowing granules. It shall be free from visible foreign matters and shall be according to the requirements specified in Table 1, when tested according to the methods prescribes in column 4 of same table.

**Table-1:
REQUIREMENTS FOR BIO ORGANO PHOSPHATE (BOP) FERTILIZER**

S. No	Characteristics	Requirements	Method of tests
01	Physical condition	Free flowing Granules	Visual inspection
02	Moisture% by weight, max	10%	Appendix – B
03	Organic P contents (P ₂ O ₅ %) by weight on dry basis (min)	20% (± 2) (Range 18 – 22%)	Appendix – C C-3
04	Organic matter % by weight on dry basis (min)	25% (± 5) (Range 20 – 30%)	Appendix – C C-4
05	Bioavailable P in soil spiked with BOP	Standard values in Table IV (Only to differentiate BOP from other fertilizers, on need basis)	Appendix – C C-5
06	Particle size	Granules (90% between 0.25 to 3 mm)	Appendix – D

3.0 PACKING AND MARKING:

3.1 **Packing:** The material shall be packed and supplied in sound, strong, moisture proof packages or container as agreed to between producer/marketer/distributor and purchaser/vendor, such as natural or synthetic fiber bags of multi wallpaper with bitumen or polyethylene moisture proofing layer. Mono film bags of heavy polyethylene (0.15-0.2 mm thickness) or in such other suitable containers. Jute or woven polypropylene bags with mono-film polyethylene liners.

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

- Name of the material, namely “Bio Organo Phosphate (BOP)”
- Minimum organic P content (as %P₂O₅)
- Name and Address of the manufacturer/importer/distributor; trade mark if any
- Net Weight in kg of the material in the container
- Any information required by law enforcement agencies or by the buyer.

3.3 Storage Requirements:

- Store in cool, clean, dry and well-ventilated area at room temperature.
- Avoid contact with moisture, as it may cause product-handling problems
- Store away from oxidizers, acids, and food and drinkable items.

4.0 SAMPLING:

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

APPENDIX – A

SAMPLING OF BIO ORGANO PHOSPHATE (BOP) FERTILIZER

A-1 GENERAL REQUIREMENTS OF SAMPLING

A-1.0 Following precautions and directions shall be observed in drawing, preparing, storing and handling test samples.

A-1.1 Samples 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/bag 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, date of sampling and other important particulars of the consignment.
- A-1.8 Samples shall be stored at 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.
- A-2.2 The number of sampling containers/bags 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/bags to be selected
N	N
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/bags 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 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 organic phosphate as P₂O₅% content shall be conducted on each of the individual test samples.

A-4.2 Test for the determination of bioavailability of P after soil incubation over a period of time shall be conducted on each of the individual samples only to differentiate BOP from other chemical phosphatic fertilizers. This test should be conducted only if required by any of the stakeholders as defined in 0.4 of foreword.

A-4.3 Test for the determination of moisture % shall be conducted on each of the individual test samples.

A-4.4 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 organic phosphate as P₂O₅% 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 individual and 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 except bioavailability of P after soil incubation, as this test is to differentiate BOP from other chemical phosphatic fertilizers and shall be conducted on special request from any of the stake holders as defined in 0.4 of foreword.

TABLE – III
CRITERION FOR CONFORMITY

S.#	Characteristic	Test Results 1,2,... n	Mean	Range	Criterion for Conformity
i.	ii.	iii	iv	V	Vi
1	Organic P ₂ O ₅ , percent by weight	--	\bar{X}	R	$\bar{x} \pm 0.6 R$ the value specified in Table (1)
2	Organic Matter	--	\bar{X}	R	$\bar{x} \pm 0.6 R$ the value specified in Table (1)

APPENDIX - B

DETERMINATION OF MOISTURE IN BOP (OVEN METHOD)

B-1 APPARATUS:

- a. Weighing bottle- size 50 mm x 30 mm, fitted with ground glass stopper with a hole.
- b. Air oven; heated electrically with temperature control system.
- c. Pestle and Mortar – made of porcelain or glass, size 102 mm (4 inch internal diameter).
- d. Balance

B-2 PROCEDURE

- B-2.1 Preparation of Sample – Place 1 gram of sample in a mortar and grind quickly particle size required is less than 1 mm, weigh 5 g of the ground sample into a weighing bottle using an analytical balance.
- B-2.2 Determination – Place the weighing bottle containing the 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 desiccator for 15 – 20 minutes. Silica gel is desirable as desiccating agent. Reweigh the sample using an analytical balance.

B-3 CALCULATION:

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

Where

A = Weight in g before heating

B = Weight in g after heating.

APPENDIX – C

ANALYSIS OF BIO ORGANO PHOSPHATE (BOP) FERTILIZER

C-1 QUALITY OF REAGENTS:

- C-1.1 Unless specified otherwise, Analytical grade chemicals and distilled water (see PS: 593-1996*) shall be used in tests.

NOTE: - 'Analytical grades Chemicals' shall mean chemicals that do not contain impurities that affect the results of analysis.

C-2 PREPARATION OF SAMPLE:

- C-2.1 Crush 50 g of BOP to pass through 500 microns PS Sieve. Dry to constant weight to obtain the prepared sample and keep in clean glass-stoppered weighing bottle in a desiccator for subsequent tests.

C-3 ANALYSIS OF ORGANIC P CONTENTS (P_2O_5) IN BOP FERTILIZER

- C-3.1 **PRINCIPLE** : Determination of organic P contents (P_2O_5) in the BOP thorough Spectro photometric method

C-3.2 APPARATUS

- Analytical Balance
- Glassware : volumetric flasks, pipettes, pipette fillers, beakers, etc.
- Spectro Photometer
- Digestion Block
- Fume Hood

C-3.3 REAGENTS (According to ICARDA manual)**C-3.3.1 Ammonium Heptamolybdate** - Ammonium Vanadate in Nitric Acid

- a. Dissolve 22.5 grams of ammonium heptamolybdate $[(\text{NH}_4)_6 \text{Mo}_7 \text{O}_{24} \cdot 4\text{H}_2\text{O}]$ in 400 mL distilled water.
- b. Dissolve 1.25 grams of ammonium metavanadate $[\text{NH}_4\text{VO}_3]$ in 300 mL hot distilled water.
- c. Add (b) to (a) in a one-liter volumetric flask, and let the mixture cool to room temperature.
- d. Slowly add 250 mL concentrated nitric acid (HNO_3) to the mixture, cool the solution to room temperature, and bring to 1-L volume with Distilled water.

C-3.3.2 Preparation of stock standard solution

- a. Dry about 2.5 g potassium di-hydrogen phosphate $[\text{KH}_2\text{PO}_4]$ in an oven at 105°C for 1 hour, cool it in desiccator and store in a tightly stopper bottle.
- b. Dissolve 0.2197 g dried potassium di-hydrogen phosphate in Distil water, and bring to 1-liter volume with distilled water.
- c. This solution contains 50 ppm P (Stock Solution).

C-3.4 Method**C-3.4.1 Digest 0.1 g dried material with sulfuric acid and hydrogen peroxide according to the method of McGill and Figueiredo (1993).**

- a. Place 0.1 g of BOP fertilizer in digestion tubes, add 2mL of concentrated H_2SO_4 and incubate digestion tubes overnight at room temperature.
- b. After incubation, pour 1 mL of H_2O_2 (35% A. R. grade extra pure) through the sides of digestion tubes and rotate.
- c. Place tubes in digestion block and heat up to 350°C until fumes are produced and continued to heat for another 30 minutes. After that remove tubes from digestion block and cool.
- d. Add 1 mL of H_2O_2 slowly. Place tubes back into digestion block until fumes are produced for 20 minutes.
- e. Repeat above process until colorless solution is obtained.
- f. Make up extract volume up to 50 mL by adding distilled water.
- g. Filter the made up volume and use for determination of total phosphorus.

C-3.4.2 Measurement of organic P contents (P_2O_5) by spectrophotometer (According to ICARDA manual)

- a. Pipette 10 mL of digested filtrate into a 100 mL volumetric flask.
- b. Add 10 mL ammonium vanadomolybdate reagent, dilute solution to volume with distilled water.
- c. Prepare a standard curve:
 - Pipette 1, 2, 3, 4, and 5 mL standard stock solution into 100 mL volumetric flasks to prepare 0.5, 1.0, 1.5, 2.0, and 2.5 ppm working standards of P. Add 10 mL ammonium- vanadomolybdate reagent in each flask and make volume up to mark with distilled water.
 - Make a blank with 10 mL ammonium-vanadomolybdate reagent and proceed as for the samples and working standards.
 - Read absorbance of blank, standards, and samples after 30 minutes at 410 nm wavelengths.
 - Prepare a calibration curve for standards, plotting absorbance against the respective P concentrations.
- d. Read organic P concentration in the unknown samples from the calibration curve.

C-3.5 Calculation of organic P contents (P₂O₅%)

$$\% \text{ Organic P contents} = \frac{\text{ppm P}}{10,000} \times 5,000 \times 2.29$$

Where :

- ppm P = reading from calibration curve
- 5,000 = Dilution factor
- 10,000 = for conversion from ppm to %
- 2.29 = Factor for converting P% to organic P contents (P₂O₅%)

C-4 ANALYSIS OF ORGANIC MATTER IN BOP FERTILIZER

C-4.1 **PRINCIPLE** : Loss of weight on ignition.

C-4.2 APPARATUS

- a. Crucible
- b. Balance
- c. Muffle Furnace
- d. Desiccator

C-4.3 Method

C-4.3.1 Weigh clean dry crucible (W₁).

C-4.3.2 Add 5 g of sample in a tarred crucible and oven dry at 105 °C for 4 hours.

C-4.3.3 Weigh sample and crucible. This yields the oven dry weight (W₂).

C-4.3.4 Place crucible with same oven dried sample in furnace at 550 to 600 °C for 6 hours to burn sample and form ash. Cool the crucible and ash in desiccator and weigh. This yields the ash weight (W₃).

C-4.4 Calculation

$$\text{Ash (\%)} = \frac{W_3 - W_1}{W_2 - W_1} \times 100$$

$$\text{Organic carbon (\%)} = 100 - \text{Ash (\%)}$$

$$\text{Organic matter (\%)} = \text{Organic carbon (\%)} \times 1.742$$

Where :

- W₁ = Weight of clean dry crucible
- W₂ = Weight of crucible and oven dry sample
- W₃ = Weight of cool crucible and ash, after furnace burning
- 1.742 = Factor for converting Organic Carbon (%) to Organic Matter (%)

C-5 DETERMINATION OF BIOAVAILABLE P IN SOIL SPIKED WITH BOP FERTILIZER

C-5.1 **PRINCIPLE:** Bioavailability of P after soil spiked with BOP fertilizer over a period ranging from 2 to 13 weeks through sodium bicarbonate extractions and colorimetric analysis.

C-5.2 APPARATUS

- a. Analytical balance
- b. UV visible Spectrophotometer
- c. Orbital shaker
- d. Centrifuge
- e. Extraction bottle, 250 mL with stopper
- f. Glassware of standardized laboratory Beakers, volumetric flasks, pipettes, funnels etc.
- g. Incubator and plastic beakers for incubation

C-5.3 REAGENTS

C-5.3.1 Sodium Bicarbonate Solution (NaHCO_3), 0.5 M. 42 grams of sodium bicarbonate is dissolved in 900 mL of distilled water. pH is adjusted to 8.5 by using 5 N NaOH solution and make the volume 1L with distilled water. Store the solution in glass or plastic container.

C-5.3.2 Sulfuric Acid Solution (H_2SO_4), 5 N. 148 mL of concentrated sulfuric acid is diluted with distilled water in the fume hood. Mix and cool it, then make the volume 1L by using distilled water.

C-5.3.3 Standard Stock Solution of Phosphorus.

- a. Dry potassium dihydrogen phosphate (KH_2PO_4) in oven at 105°C for an hour and is cooled in desiccator and store it in airtight bottles.
- b. Weigh 2.197 g (KH_2PO_4) and dissolve in to a 1L volumetric flask by using distilled water and the concentration of this stock solution is 500 ppm
- c. Take 50 mL from stock solution into a 250 mL volumetric flask and make the volume by using distilled water, the concentration of this sub stock is 100 ppm
- d. Now prepare working standards of 2, 4, 6, 8, 10, 12, 14, 16 and 18 ppm by dissolving 2, 4, 6, 8, 10, 12, 14, 16 and 18 mL in 100 mL volumetric flask from sub stock solution (100 ppm).

C-5.3.4 Color Developing Reagent (CDR)

- a. Reagent A
 - Dissolve 12 g of ammonium heptamolybdate ($(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$) in 250 mL of distilled water.
 - Dissolve 0.2908 g of antimony potassium tartrate ($\text{KSbO}\cdot\text{C}_4\text{H}_4\text{O}_6$) in 100 mL of distilled water.
 - Add and dissolve both reagents in a 2 L volumetric flask. Also add 5 N Sulfuric acid solution, mix it well and make the volume with distilled water and keep away from light and heat.
- b. Reagent B
 - Dissolve 1.056 g of L. ascorbic acid ($\text{C}_6\text{H}_8\text{O}_6$) in 200 mL of reagent A and mix thoroughly. Reagent B is prepared according to the need as it becomes ineffective after 24 hours.

C-5.4 PROCEDURE

- a. Take six samples of 500 g sieved soil in separate plastic beakers. Label three beakers as "Fertilizer", three beakers as "Soil" and make three replications. Spike the soil of beakers labeled as "Fertilizer" with 100 g of BOP, any other P fertilizer or any other material that needs to be checked. The beakers labeled as "Soil" should be maintained without addition or spiking with any fertilizer material. Maintain soil water holding capacity at 50%. Incubation should be carried out for 13 weeks at $28 \pm 1^\circ\text{C}$.

- b. Take 5 g well mixed soil sample from each beaker periodically at 7 days interval.
- c. Transfer air-dried soil samples in separate flasks of 250 mL each and add 100 mL of sodium bicarbonate (NaHCO_3) solution (0.5 M) with pH 8.5 in each flask.
- d. Shake each flask at 200 – 300 rpm for 30 minutes.
- e. Filter each solution, separately, by using filter paper Whatman No. 42.
- f. Transfer 10 mL of each filtrate in separate 100 mL volumetric flasks and add 10 mL of reagent B and make the volume 100 mL with distilled water in each flask.
- g. Shake each flask well and take readings against absorbance by feeding; first blank, then Standards, followed by 6 samples of “*Fertilizer*” and “*Soil*”.
- h. Make calibration curve for Standards by plotting absorbance against P concentrations.
- i. Measure P concentration of “*Fertilizer*” and “*Soil*” samples from the calibration curve.

$$\text{Extractable Phosphorus (ppm)} = \text{ppm P from calibration curve} \times \frac{A}{W_t} \times \frac{50}{V}$$

Where

A = Total of the extract (mL)

W_t = Weight of air-dried soil (g)

V = Volume of extract used for analysis

50 = Volume made after color development

- j. Calculate average extracted P from “*Fertilizer*” and “*Soil*” (6 samples, 3 replications); and record average of “*Fertilizer*” under column A and of “*Soil*” under column B in table IV.
- k. Calculate Bioavailable P (ppm) as per following formula.
= Average extracted P from “*Fertilizer*” – Average extracted P from “*Soil*” samples
- l. Compare Bioavailable P (ppm) value of column C in table IV with corresponding standard value given in column D. If values of column C and D are in close proximity then the product will be BOP, if not then it is either chemical fertilizer or some other material.
- m. This test may be continued for minimum of 2 weeks to maximum of 13 weeks until the type of product is clearly identified by the testing authority. If values of column C follows the trend of values given in column D then the product is BOP if not then it is either chemical fertilizers or some other material.

Table IV : Comparison of Bioavailable P (ppm)

Weeks	Bioavailable P (ppm) calculation			Standard values
	Average extracted P (ppm) from spiked soil (" <i>Fertilizer</i> ")	Average extracted P (ppm) from soil without spiking (" <i>Soil</i> ")	Bioavailable P (ppm) from spiked soil	Bioavailable P (ppm) from soil spiked with BOP
	A	B	C = A - B	D
1				150 ± 2
2				170 ± 2
3				190 ± 2
4				190 ± 2
5				210 ± 2
6				210 ± 2
7				210 ± 2
8				270 ± 2
9				290 ± 2
10				300 ± 2
11				330 ± 2
12				340 ± 2
13				350 ± 2

APPENDIX-D**DETERMINATION OF SIZE DISTRIBUTION OF BOP****D-1 APPARATUS / EQUIPMENT**

- D-1.1 *Stainless steel sieves* of the required mesh size with lid & bottom pan.
- D-1.2 *Sieves Shaker*
- D-1.3 *Balance*
- D-1.4 *Brush*

D-2 METHOD:

- D-2.1 Arrange the individually tare sieves in descending order of mesh size from top to bottom.
- D-2.2 Place receiving pan on the bottom of stack
- D-2.3 Weigh about 200 to 300g of sample taken through sample divider.
- D-2.4 Transfer sample onto the top sieve place lid on top of stack.
- D-2.5 Place the sieve stack on shaker and tighten the belts evenly on both sides.
- D-2.6 Set timer of vibrator to 5 minutes amplitude at 3.0 mm and start the vibrator.
- D-2.7 After shaking stops switch-off the vibrator and remove the sieves one by one.
- D-2.8 Weigh sieve + sample on top loading balance.
- D-2.9 Calculate the weight of samples retained on each sieve.

D-3 CALCULATION

- D-3.1 Calculate weight percent on each sieve by following formula

$$\text{Weight \%} = \frac{\text{Weight (g) on sieve}}{\text{Total Weight of Sample}} \times 100$$