

---

# **PAKISTAN STANDARD**

---

## **SPECIFICATION FOR POTASSIUM SULPHATE (FERTILIZER GRADE)**



---

(All Rights Reserved)

**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.**

**PAKISTAN STANDARDS SPECIFICATION**  
**FOR**  
**POTASSIUM SULPHATE (SULPHATE OF POTASH)**  
**FERTILIZER GRADE, (2<sup>nd</sup> Revision)**

**0. FOREWARD**

- 0.1 This Pakistan Standard (Second Revision) was adopted by Standards Development Centre; PSQCA on 26<sup>th</sup> October, 2011 after the draft finalized by the Fertilizers and Allied Product Technical Committee had been approved by the Chemical National Standards Committee.
- 0.2 The Pakistan Standard Specification was first established in 1968 and first revised in 1983, keeping in view the latest developments made in the industries; the committee felt it is necessary to further revise and update. The Technical Committee revises the limit of total chlorides (as Cl) and included the Sulfur and sodium contents in requirement Table-1 to harmonizing the Pakistan Standard with International Standards.
- 0.3 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. Furthermore, due weightage had to be given to the need for international co-ordination among standard prevailing in different countries of the world.
- 0.4 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.
- 0.5 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 Technical Committees for consideration at the time of revision.
- 0.6 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 describes the requirements, methods of test and sampling of Potassium sulphate for used as fertilizer. The material is also known as sulphate of Potash (SOP).

**5. REQUIREMENT:**

- 5.1 The material shall consist essentially of potassium sulphate  $K_2SO_4$  and shall be in the form of free-flowing crystalline powder or granules. It shall be free from visible, foreign matters and shall comply with the requirements specified in Table – 1, when tested according to the methods prescribes in column – 4 of Table -1

**Table-1**  
**REQUIREMENTS FOR POTASSIUM SULPHATE (FERTILIZER GRADE)**

S. No	Characteristics	Requirements	Method of tests
01	Physical condition	Free flowing granular or crystalline	Visual inspection
02	Moisture% by weight, max	1.0	Appendix – B
03	Potassium content( as K <sub>2</sub> O % by weight) min.	50.0	Appendix – C
04	Total chlorides (as Cl), % by weight), (on dry basis) max.	2.5	Appendix – C C- 5
05.	Particle size	90% between 0.25 to 3mm.	Appendix – D
06	Sodium (as NaCl) % by weight, max	2	Appendix – E
07	Sulfur % by weight, min.	17.5	Appendix – F

6. **SAMPLING:**

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

7. **PACKING AND MARKING:**

7.1 **Packing:** - 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 a 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 can also be used.

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

- (a) Name of the material, namely “Potassium sulphate, Fertilizer Grade or “Sulphate of Potash (SOP)
- (b) The minimum potassium content (as K or K<sub>2</sub>O) 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 container
- (e) Any information required by law enforcement agencies or by the buyer.

7.3 **Storage:** Store in cool, clean dry and well ventilated area. Avoid contact with moisture, as it will cause product handling problem. Store away from oxidizers, acids and from food or drink.

## APPENDIX – A

### SAMPLING OF POTASSIUM 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 potassium content shall be conducted on each of the individual test samples.

A-4.2 Test for the determination of Total Chlorides shall be conducted on each of the individual test samples.

A-4.3 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 potassium 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 1,2.... n	Mean	Range	Criterion for Conformity
i.	ii.	iii	iv	v	vi
1	Total potash, percent by weight	--	$\bar{X}$	R	$\bar{x}-0.6 R \geq$ the value specified in Table (1)

**APPENDIX - B**

**DETERMINATION OF MOISTURE (OVEN METHOD)**

**B-1 APPARATUS:**

B-1.1 Weighing bottle- size 50 mm x 30 mm, fitted with ground glass stopper with a hole.

B-1.1.1 Air oven; heated electrically with temperature control system.

B-1.1.2 Mortar – made of porcelain or glass, size 102 mm (4 inch internal diameter).

**B-2 PROCEDURE**

B-2.1 Preparation of Sample – Place 1 gram of sample in a mortar and grind quickly particle size required, less than 1 mm, weigh 5 gram 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:**

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

Where

A = Weight in gram before heating

B = Weight in gram after heating.

**APPENDIX – C****C-0 ANALYSIS OF POTASSIUM SULPHATE, FERTILIZER GRADE****C-1 QUALITY OF REAGENTS:**

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

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

**C-2 PREPARATION OF SAMPLE:**

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

**C-3 DETERMINATION OF TOTAL POTASSIUM****C-3.1 PRINCIPLE**

C-3.1.1 Determination of Total Potassium in (SOP) through flame photometric method

**C-3.2 APPARATUS**

- Analytical Balance
- Volumetric flasks
- Flame photometer
- Pippets
- Pippet filler
- Wash bottles
- Glass Beaker

**C-3.3 REAGENTS****C-3.3.1 Preparation of Stock Standard Solution (1mg K<sub>2</sub>O/mL)**

Dissolve 2.889 g dried (02 Hrs. at 105<sup>0</sup>C) KH<sub>2</sub>PO<sub>4</sub> in H<sub>2</sub>O, and dilute to 1L.  
(1mL= 1mg K<sub>2</sub>O)

**C-3.3.2 Preparation of Working Standard Solution (10, 20,30,40,50 &55 ppm K<sub>2</sub>O)**

Accurately measure by burette 10 , 20 & 30mL stock standard solution into 1l volumetric flask to give 10, 20 & 30 PPM K<sub>2</sub>O and 20, 25 & 27. 5mL into 500ml volumetric flask to give 40, 50, & 55 PPM K<sub>2</sub>O solution.

**C-3.3.3 Preparation of Standard Curve**

Prepare curve for 55ppm K<sub>2</sub>O by aspirating working solution 2.2.

**C-4 PROCEDURE**

C-4.1 Prepare a solution using 1g test portion in 1L demin or distil water and filter if required

C-4.2 Transfer 25mL test solution to 250mL volumetric flask, dilute to volume with H<sub>2</sub>O and mix.

- C-4.3 Atomize portions of test solution several times to obtain reliable average readings for each test solution.
- C-4.4 Determine PPM K<sub>2</sub>O from standard curve 2.3.
- C-4.5 Calculate percent K<sub>2</sub>O as follows:

C-5 **CALCULATIONS:**

$$\% \text{K}_2\text{O} = \frac{\text{PPM K}_2\text{O} \times 1000 \times 250}{\text{Wt of Sample} \times 25 \times 10000}$$

C-6 **DETERMINATION OF TOTAL CHLORIDES**

C-6.0 **Principle of method** –Determination of Total Chloride in (SOP) thorough flame photometer method.

C-6.1 **APPARATUS:**

Titration flask	:	250 mL
Pipette	:	2mL, 10mL
Burette	:	25 mL
Cylinder	:	50mL, 100mL
Volumetric flask	:	1L

C-6.2 **REAGENTS**

C-6.2.1 **Silver Nitrate (0.05 N)**

Dissolve 8.4935 AgNO<sub>3</sub> crystals in demin water and dilute to 1 liter. Standardize against the 0.05N NaCl solution using Potassium Chromate indicator. End point is appearance of brick red color.

C-6.2.2 **Sodium chloride standard solution (0.05 N)**

Dissolve 2.9225g of NaCl (previously dried at 105<sup>0</sup>C for one hr.) and dilute to 1 liter.

C-6.2.3 **Potassium Chromate Indicator**

Dissolve 5g Potassium chromate in 100mL demin water, and add AgNO<sub>3</sub> until a slight red precipitate is produced. Allow the solution to stand for atleast 24 hrs. After the addition of AgNO<sub>3</sub>. Then filter the solution.

C-6.2.4 **Phenolphthalein Indicator**

Dissolve 0.1g of solid phenolphthalein in 100mL of 95% ethanol.

C-6.2.5 **H<sub>2</sub>S<sub>4</sub>O (0.02 N)**

- Dilute 200mL of conc. 0.1 N H<sub>2</sub>SO<sub>4</sub> to 1 liter
- standardized against 0.02 N Na<sub>2</sub>CO<sub>3</sub> solution using methyl red indicator. End point is from yellow to bright red. Near end point slightly warm the solution and then titrate till appearance of bright red color.

C-6.2.6 **NaOH (0.02 N)**

Dilute 10mL of 0.5 N NaOH to 250mL.



**C-6.3 METHOD**

C-6.3.1 Dissolve 1g MOP sample in water and mix to dissolve. Filter if required and make volume up to 1L in volumetric flask.

C-6.3.2 Take 100mL filtered sample or an aliquot diluted to 100mL in titration flask and add 3-4 drops of Phenolphthalein indicator.

C-6.3.3 If solution turns pink, neutralize it with 0.02 N H<sub>2</sub>SO<sub>4</sub>, but if solution remains colorless, first make alkaline with 0.02 N NaOH and then neutralize with 0.02 N H<sub>2</sub>SO<sub>4</sub> (pH of solution should be 7-8.3).

C-6.3.4 Add 1mL Potassium chromate indicator and shake.

C-6.3.5 Titrate against 0.05 N Silver Nitrate until brick red color appears. Verify by adding one more drop.

C-6.3.6 Note volume of 0.05 N AgNO<sub>3</sub> consumed as "V" mL.

**C-6.4 CALCULATION:**

$$\text{Chloride ( \% as Cl)} = \frac{\text{"V"} \times 0.05 \times 35.45 \times 1000 \times 100}{\text{Wt of MOP} \times \text{mL of Sample} \times 1000}$$

**APPENDIX – D**  
**DETERMINATION OF SIZE DISTRIBUTION OF POTASSIUM SULPHATE**

**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 *Top Loading 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 weigh percent on each sieve by following formula

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

## APPENDIX – E

### DETERMINATION OF SODIUM

- E-1 **Principle of Method-** Determination of Sodium in (SOP) through flame photometer method.

### E-2 **APPARATUS**

- E-2.1 Flame Photometer  
E-2.2 Volumetric Glass Apparatus

### E-3 **REAGENTS**

#### E-3.1 **Preparation of Standard Solution (1000ppm Na)**

Dry solid A.R grade NaCl at 105<sup>0</sup>C for 02 Hrs. Dissolve 2.54g NaCl in distil or demin water and make volume up to 1L. (1ml = 1mg Na).

#### E-3.2 **Preparation of Working Solution “A” (0~40ppm Na)**

Prepare a range of dilution 0~40ppm Na in 250 mL volumetric flask from above standard solution with intervals ≤ 5ppm and up to full scale of 40ppm Na.

#### E-3.3 **Preparation of Working Solution “B” (0~10ppm Na)**

Prepare a range of dilution 0~10ppm Na in 250 mL volumetric flask from above standard solution with intervals 2ppm and up to full scale of 10ppm Na.

#### E-3.4 **Preparation of Standard Curve for Sample containing 1% or more Na**

Prepare curve for 0~40ppm Na by aspirating working solution “A”.

#### E-3.5 **Preparation of Standard Curve for Sample containing less than 1% Na**

Prepare curve for 0~10ppm Na by aspirating working solution “A”.

**E-4 PROCEDURE**

- E-4.1 Prepare a solution using 2.5g test portion (<4% Na) or 1.25g (4~20% Na) in 250ml demin or distil water and filter if required.
- E-4.2 Transfer 25 mL (<4% Na) or 10 mL (4~20% Na) test solution to 250mL volumetric flask, dilute to volume with H<sub>2</sub>O and mix.
- E-4.3 Atomize portions of test solution several times to obtain reliable average readings for each test solution.
- E-4.4 Determine PPM Na from standard curve "A" or "B"
- E-4.5 Calculate percent Na as follows:

**E-5 CALCULATION:**

0~4%: PPM Na /10 = percent Na

4~20%: PPM Na /2 = percent Na

**APPENDIX – F****DETERMINATION OF SULPHUR IN POTASSIUM SULPHATE****F-1 APPARATUS/ EQUIPMENT**

- F-1.1 Volumetric flasks : 1L, 250 ml
- F-1.2 Beaker : 400 ml, 250 ml
- F-1.3 Porcelain
- F-1.4 Muffle furnace
- F-1.5 Whatman filter paper # 41 (medium porosity)

**F-2 CHEMICAL/REAGENTS:**

- F-2.1 Barium Chloride Solution
- F-2.2 Dissolve 122g of BaCl<sub>2</sub> in 500ml demin water contained in 1L volumetric flask. Mix to dissolve and make the volume upto mark.

**F-3 HCL (Conc.)**

**F-4 METHOD**

- F-4.1 Weigh 4~5g of sample in 400ml beaker and add 100ml demin / distil water.
- F-4.2 Transfer the contents into 250ml volumetric flask.
- F-4.3 Make the volume upto mark with demin / distil water. Mix well and filter 100ml through whatman-41 filter paper.
- F-4.4 Take 20ml of filtrate in 250ml beaker. Add 100ml demin distil water and 2ml HCl (conc).
- F-4.5 Boil the solution for few minutes.
- F-4.6 Add 50ml BaCl<sub>2</sub> Solution and keep boiling for further few minutes.
- F-4.7 Cover beaker with clock glass and place in water bath for 1 hours at 80<sup>0</sup>C.
- F-4.8 Decant the liquid through ash less filter paper whatman-41 qualitatively, if necessary.
- F-4.9 Wash the ppt. on filter paper with demin / distil water until washings become chloride free (check precipitation with AgNO<sub>3</sub> solution).
- F-4.10 Place filter paper in porcelain crucible previously conditioned (at 800<sup>0</sup>C) and weighed "W<sub>1</sub>" g.
- F-4.11 dry and ignite in porcelain for one hour at 800<sup>0</sup>C in furnace (pre burn the filter on flame to avoid formation of excessive fumes in furnace).
- F-4.12 Cool in desiccator and weigh as "W<sub>2</sub>" g.

**F-5 CALCULATION**

$$\text{Sulfur \%} = \frac{W_2 - W_1 \times 250 \times 32 \times 100}{\text{Wt. of sample (from step 3.1)} \times 20 \times 233}$$

**REFERENCES:**

In the preparation of this standard references was made to the following:

- *Pakistan Standard PS: 582- 1996, Glossary of terms used in Fertilizer Industry (1<sup>st</sup> Revision)*
- *Encyclopedia of Chemical Technology, 4<sup>th</sup> edition, Vol: 10, John Wiley & Sons Inc. (pg: 449-451)*
- *MS 16 Specification for Potassium sulphate (Fertilizer Grade)*
- *IS: 2764 Specification for Potassium sulphate (FG)*
- *AOAC-2.5.04 17<sup>th</sup> Edition determination of Potassium in Fertilizer by flame photometer method*
- *AOAC-2.6.09 17<sup>th</sup> Edition determination of Chloride in Fertilizer by flame photometer method*
- *AOAC-2.6.28 17<sup>th</sup> Edition determination of Sulphur in Fertilizer by flame photometer method*