



# Institute for Industrial Research & Toxicology

## औद्योगिक अनुसंधान एवं विष विज्ञान संस्थान

Registration No. 1303/C/CPCSEA (Ministry of Environment & Forests, Government of India)  
License No.: 01/2015 (Food and Drug Administration, UP)  
AN ISO 9001 : 2015, ISO 14001 : 2015, ISO 45001 : 2018 Certified Organization  
GLP Certified, NABL (ISO/IEC 17025) Accredited

### DETERMINATION OF ACCELERATED STORAGE STABILITY OF POTASSIUM SALT OF ACTIVE PHOSPHORUS

(Protocol adopted- (CIPAC)MT- 46.1.3)

#### MANUFACTURER & SPONSOR

ISHA AGRO INDIA

#### STUDY PERFORMED BY

INSTITUTE FOR INDUSTRIAL RESEARCH AND TOXICOLOGY

F-209, UPSIDC, PHASE-I, MG Road,

Ghaziabad -201302, Uttar Pradesh

Project No. : CH210EM293

Report No. : IIRT/CHEM/151607

Date: 25/04/2018



TEST COMPOUND : Potassium Salt of Active Phosphorus  
 SPONSORED BY : ISHA AGRO  
 TEST : DETERMINATION OF ACCELERATED STORAGE STABILITY  
Project No. : CH210EM293  
Report No. : IIRT/CHEM/151607



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**STATEMENT OF COMPLIANCE**

The study Storage Stability of Potassium salt of Active Phosphorus Entitled “**Determination of Accelerated Storage Stability**” was performed at Institute for Industrial Research & Toxicology, Ghaziabad.

I hereby attest the authenticity of the study and guarantee that this report represents a true and accurate record of the result obtained.

The study was conducted as per Protocol adopted in- (CIPAC)MT- 46.1.3

All the original raw data including electronically record data, documentation, the signed plan, draft report, copy of final report and the representative test items are archived in the archives at Institute for Industrial Research & Toxicology, Ghaziabad.

I, the undersigned, take overall responsibility for the reliability of the work described in the report “Determination of Accelerated Storage Stability” with compliance of Good laboratory Practice for Storage Stability of test sample.



**NN Mishra**  
**Director/Head technical**  
**Date: 25/04/2018**

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### GLP STATEMENT OF COMPLIANCE

**Study Title: “Accelerated Storage Stability test of Potassium salt of Active phosphorus  
”(Determination of potash and Phosphorus content)”**

This study was conducted according to Good Laboratory practice principles as published by OECD 1998, number 1 [ENV/MC/CHEM (98) 17].

This study was conducted in accordance with the written protocol, authorized by the sponsor and IIRT management and following the standard operating procedures of IIRT.

There were no known circumstances that may have affected the quality or integrity of the data.

All raw data, documentation, the protocol, protocol amendments, the final report and 1 g sample of the test substance are retained in the GLP Archives at IIRT, Ghaziabad.

We, the undersigned, take overall responsibility for the reliability of the work described in the report “Determination of Accelerated Storage Stability” with compliance of Good laboratory Practice for Physico-chemical properties of test sample.



**Susheel kumar**

**Analytical chemist**

**Date : 25/04/2018**



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### QUALITY ASSURANCE STATEMENT

This is to certify that the study entitled “**Analysis of Storage Stability of Potassium salt of Active Phosphorus (Determination of Potassium salt of Active Phosphorus)**” has been audited and the final report examined with respective raw data. The report is true reflection of raw data and guidelines (OECD). The study was conducted in compliance with Good laboratory practice.

Finding results from the audits were reported to the study director and management on the dates specified below.

Phase of study	Date of audit
Protocol	05/04/2018
Study audit	06/04/2018
Draft report	25/04/2018

Inspections were performed according to the standard operating procedures of the test facility’s Quality Assurance unit. The report was inspected against the approved study plan and pertinent raw data and found to accurately reflect the same.

Date: 25/04/2018



Shalini Mishra

For the Quality Assurance Unit

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**STUDY PERSONNEL**

**Senior Scientist (Chemistry)** : Susheel kumar M.Sc.

**Senior Analytical Chemist** : Atul Kumar B.Sc.

**Analytical Chemist** : Ankur Rana B.Pharma

<b>TEST COMPOUND</b>	:	<b>Potassium Salt of Active Phosphorus</b>
<b>SPONSORED BY</b>	:	<b>ISHA AGRO</b>
<b>TEST</b>	:	<b>DETERMINATION OF ACCELERATED STORAGE STABILITY</b>
<b><u>Project No.</u></b>	:	<b>CH210EM293</b>
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### SUMMARY

Accelerated Storage Stability study of Potassium salt of Active Phosphorus (Supplied by M/S Isha Agro, the study was conducted at Institute for Industrial Research & Toxicology, Ghaziabad by storing the samples at  $54 \pm 2^{\circ}\text{C}$  temperature for a period of 14 days following standard protocol and good laboratory practices.

The test sample was analysed for physico-chemical property acidity and potash and Phosphorus Active ingredient content study on day 0 (Pre storage) and on day 14<sup>th</sup> (Post storage).

The result obtained from present investigation showed that the analysis of the test samples showed no significant change in active ingredient content after 14 days of storage period. Furthermore, the sample results are under the nominal value before and after the study period. Results showed that there was under limit change in the active ingredient and other physico-chemical properties when stored at 14 days after keeping the test sample under prescribed temperature. Thus from the above experimental results, it could be concluded that Potassium salt of Active Phosphorus is stable on storage on elevated temperature of  $54 \pm 2^{\circ}\text{C}$  for 14 days.

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**GENERAL INFORMATION**

1.	Common name	:	Potassium Salt of Active Phosphorus.
2.	Chemical Name	:	Phosphonic acid, monopotassium salt phosphonic acid.
3.	Synonyms		Monopotassium salt of active phosphorus acid, Potassium dihydrogen phosphate, monopotassium phosphate, monopotassium phosphate. dipotassium salt of phosphorus acid, dipotassium hydrogen phosphite, dipotassium phosphite, dipotassium phosphonate.
4.	Empirical formula	:	$\text{KH}_2\text{PO}_4$ , $\text{K}_2\text{HPO}_4$
5.	CAS Number	:	13977-65-6 13492-26-7 7758-99-8
6.	Molecular weight	:	136.08 g/mol
7.	Method follow	:	CIPAC MT-46.1.3



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**TEST ITEM DETAILS**

1.	Name of the test substance	:	Potassium salt of Active Phosphorus.
2.	Batch No.	:	001
3.	Date of Mfg.	:	Jan 2018
4.	Date of Exp.	:	Dec 2021
5.	Purity	:	1.Potassium - 2.Phosphorus -
6.	Name and address of the sponsor	:	M/S Isha Agro
7.	Name and address of the test facility	:	Institute for industrial research and toxicology, F-209, UPSIDC, MG Road, Ghaziabad,

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## MATERIALS AND METHODS

### OUT LINE OF METHOD

A Sample is placed in a glass bottle, which is subsequently closed and placed in an oven at a specified temperature for a defined period of time.

### Apparatus

- (a) *Oven* flameproofed and thermostatically maintained at the required temperature ( $\pm 2^{\circ}\text{C}$ ).
- (b) *Glass bottles* 100 to 125 ml fitted with screw caps and polyethylene inserts.

### Procedure

1. Before the study test the sample as per specification
2. Pour 50gm of sample in a bottle, fit the polyethylene insert and and put the uncapped bottle and contents in the oven for 0.5 h and afterwards packed the bottle with screw cap making sure there should not be any chances of leakages.
3. Keep it in an oven at  $54 \pm 2^{\circ}\text{C}$  under unique temperature for a period of 14 days.
4. Remove the bottle from the oven after completion of 14 days and remove the cap, and allow the bottle and contents to cool naturally to room temperature, replacing the cap when cool.
5. Carry out the appropriate tests specified in the general methods of analysis for emulsifiable concentrates within 24 h of cooling.
6. Calculate active ingredients and other parameter before and after the study and compare it.



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**ANALYSIS OF PHOSPHORUS ACTIVE INGREDIENT CONTENT**

**Scope :** This method prescribes Titrimetric method for determination of phosphorus and applicable to determined phosphorus when present in higher percentage.

**Theory:** When a solution of Orthophosphoric Acid is treated with sodium molybdate and quinoline, quinoline molybdate is precipitated which can be isolated and titrated with std. sodium hydroxide.

**Interferences:** Ammonium salts interferes and must be eliminated by means of sodium nitrite or hypobromide treatments. Sulphuric acid leads to high and erratic results and it should not be used.

**Sample Preparations:** weighed about 1mg nearest to 0.1mg in digestion flask,dissolved and treated with 25ml concentrated Hydrochloric acid or concentrated Nitric acid by refluxing for atleast 1/2hour.After cooling the volume is made to 100ml.

**Apparatus / Equipments involved:**

1. Burette -100ml
2. Pipette – 50ml
3. Beaker – 1000ml
4. Standard flask – 1000ml
5. Measuring cylinder – 50ml
6. Digestion flask – 250ml
7. Heating mantle / Hot plate
8. Magnetic stirrer.

**Reagents Required:**

1. Distilled water
2. Sodium molibdate Solution 15%
3. Quinoline hydrochloride solution – took 800ml distilled water in 1000ml glass beaker, 25ml Conc.Hcl carefully added and boil it then added slowly 20ml of re-distilled quionoline, stir well and allow it to room temperature. Filter with suction pump by using paper pulp pad but don't wash it, made up the volume to 1000ml.
4. Mixed indicator solution: Mix two volumes of 0.1% phenolphthalein solution and three volumes of thymol blue solution (Both in ethanol)

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**Procedure:**

1. Took 100ml sample solution in a conical flask and added 30ml sodium molybdate 15% solution.
2. Heat to boil again and added 60ml of quinoline molybdate solution drop by drop from burette with swirling the solution to continuous boiling.
3. The coarsely-crystalline precipitated is obtained and allow it to stand in water bath for 15min and then cold to room temperature.
4. Prepare a paper pulp filter in a buchner funnel and tamp well down.
5. Decant the clear solution through the filter and wash the ppt twice by decantation with 1:9Hcl. This remove most of the excess quinoline and of molybdate.
6. Wash the ppt with 30ml portions of water by decantation, six times and transfer the ppt to the same flask, washed the precipitated until the washings are acid free.
7. Added 50ml of water and stir well when the filter paper completely tear and then added 50ml of 0.5N Carbonate free sodium hydroxide solution with swirling the solution

**Calculation Formula: 01**

Phosphorus ( P<sub>2</sub>O<sub>5</sub>) Content =

$$\frac{0.03207 \times (\text{Weight of precipitate}) - (\text{Weight of blank}) \times 250 \times 100}{10 \times \text{Weight of sample}}$$

Where, 0.03207 (Factor)

10 (Dilution Factor)

**Calculation Formula: 02**

Potassium (K<sub>2</sub>O) content =

$$\frac{\text{Reading of sample} \times (\text{Vol. of sample sal}^n \text{ for analysis}) \times (\text{Volume of sample (stalk)} \times 94.196 \times 100}{\text{Slope of calibration curve} \times 1000 \times (\text{ml of sample used for working soln.}) \times 78.196 \times 107.7}$$

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### Active Content Result of 0<sup>th</sup> Day

The active ingredient content of **Phosphorus** and **Potassium** In sample **Potassium Salt of Active Phosphorus** sponsored by Isha Agro were found **40.62 & 40.76** percent by mass before storage at 54 °C on 0 day period.

### Active Content Result after 14<sup>th</sup> day

The active ingredient content of **Phosphorus** and **Potassium** In sample **Potassium Salt of Active Phosphorus** sponsored by Isha Agro were found **40.58 & 40.73** percent by mass before storage at 54 °C after 14<sup>th</sup> day period.

## Determination of pH Value of Potassium Salt of Active Phosphorus

### Experimental Method

#### Requirements

#### Reagents

- Buffer Solutions: pH 7, pH 4 and pH 10.
- Water: Distilled or deionized water:
- pH Meter: Capable of at least a two-point calibration.
- pH Electrode System: e.g. a single or dual glass electrode system conditioned and maintained according to the manufacturer's instructions.
- Beakers: 200 to 250 mL (or other suitable containers for titration)

#### Calibration:

1. Operate the pH meter and the pH electrode system according to the manufacturer's operating instructions.
2. Calibrate the measurement system (i.e. pH meter and pH electrode system) according to the manufacturer's operating instructions using at least two appropriate buffer solutions.

#### pH Measurement of a Diluted (1%) Formulation:

1. Weigh 1.0 g of sample into a mixing cylinder containing ~50 mL reagent water.



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2. Add reagent water to 100 mL, stopper and shake vigorously until the formulation is completely mixed or dispersed.
3. Transfer the solution or dispersion to a 200-mL beaker and allow any suspended material to settle for 1 minute.
4. Ensure that the temperature of the formulation/water mixture does not differ from the reference solutions used for calibration.
5. Immerse the electrode into the formulation/water mixture and measure the pH without stirring. Record the pH value after 1 minute. If the pH value changes more than 0.1 pH unit during this equilibration time, record the pH 10 minutes after immersion of the electrode.

Immerse the electrode into the formulation/water mixture and measure the pH without stirring. Record the pH value after 1 minute. If the pH value changes more than 0.1 pH unit during this equilibration time, record the pH 10 minutes after immersion of the electrode.

#### **pH at 0<sup>th</sup> Day**

The pH of Potassium Salt of Active Phosphorus, *Sponsored By Isha Agro* was found **6.57** of 1% solution.

#### **pH at 14<sup>th</sup> Day**

The pH of Potassium Salt of Active Phosphorus, *Sponsored By Isha Agro* was found **6.55** of 1% solution.

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**Final Observation:**

**Observation of the results before the study (0) day (Table 01)**

S.No	Characterization	Result
1.	Description	Material is in the farm of crystalline powder
2.	Phosphorus as P <sub>2</sub> O <sub>5</sub> content	40.62%
3.	Potassium as K <sub>2</sub> O content	40.76%
4.	pH	6.57

**Observation of the results after the study (14) day (Table 02)**

S.No	Characterization	Result
1.	Description	Material is in the farm of crystalline powder
2.	Phosphorus as P <sub>2</sub> O <sub>5</sub> content	40.58%
3.	Potassium as K <sub>2</sub> O	40.73%
4.	pH	6.55

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## **CONCLUSION**

The Potassium Salt of Active Phosphorus is stable as it comply all limits after storage at 54°C for 14 days.

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### **ARCHIVES**

The raw data, sample of the test substance, study report and other materials are being retained for one year at Institute for Industrial Research and Toxicology, Ghaziabad after completion of the study.

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**CERTIFICATE OF ANALYSIS**



**Institute for Industrial Research & Toxicology**

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**CERTIFICATE OF ANALYSIS**

NAME AND ADDRESS OF THE MANUFACTURE/SPONSOR M/s. Isha Agro India Office No. 05, Malti Complex, 4/121, Ideal Colony, Poud Road – Pune, Maharashtra, India.	Report No.	IIRT/1819/1370	Date	07-06-2018
	Party Ref.		Date	
*SAMPLE NOT DRAWN BY IIRT* Mfd. By: As above	Product Name	Potassium Salt of Active Phosphorus		
	Trade Name	PSAP		
	Sample code		ICS code	CHEM-1370
	Sample Quantity	200gm	Recd. Dt.	04-04-2018
	Mfg. Dt.	Jan-2018	Exp. Dt.	Dec-2021
	Batch No.	001	Pack cond.	Sealed
	Smp. Draw By			

**RESULT OF ANALYSIS AND PROTOCOLS OF TEST APPLIED**

Description: The material in the form of white free flowing powder, free from visible impurities

S.No.	Parameters	Method	Results	Unit
1.	Phosphorus as P <sub>2</sub> O <sub>5</sub>	FCO	40.62	%
2.	Potassium as K <sub>2</sub> O	FCO	40.76	%

Note: The above performed tests comply and confirm as per specifications.

*[Signature]*  
28-06-2018

Reported by:  
Sign/date

*[Signature]*  
28-06-2018

Reviewed by:  
Sign/date

*[Signature]*  
28-06-2018

Approved by:  
Sign/date

Note: 1) This certificate refers to only to the particular sample submitted for Testing. 2) This certificate not is produce, except in full, without the permission from the Q.M./Director of IIRT. 3) Results reported valid at the time of Testing. 4) Laboratory Standards are traceable to Nation Standard. 5) This report issued based on the Chemical Composition provided by the Sponsor.



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