

PESTICIDE FORMULATION & RESIDUE ANALYTICAL CENTRE, PMD, NIPHM, HYDERABAD

Sr. No. in Scope

NABL / NON NABL

Flow chart for analysis of Aluminium Phosphide in formulation

		Date of Analysis	
Sl. No.	Step	Execution	Executed By
1.	Sample No.		
2.	Name of Sample		
3.	Preparation of Standard 0.5 N Potassium Permanganate solution :		
3.1	Weigh 15.8 g of Potassium permanganate (KMnO ₄) in a 250 mL beaker.	g	
3.2	<i>Note down the S.No. of balance log book.</i>		
3.3	Dissolve and quantitatively transfer with 1000 mL distilled water to an amber coloured bottle to get 0.5 N solution.		
4.	Preparation of 0.5 N Oxalic acid Solution :		
4.1.	Weigh accurately 15.75 g of AR grade oxalic acid on an analytical balance in a 250 mL beaker and dissolve in about 200ml of water.	g	
4.2	<i>Note down the S.No. of balance log book.</i>		
4.3	Transfer the solution (4.1) to a 500 ml volumetric flask. Add 125ml of sulphuric acid 1:1 (v/v) solution and make up the volume with water .		
5	Standardization of 0.5 N KMnO₄ Solution:		
5.1	Pipette out in 25ml of 0.5 N oxalic acid solution into a 250 mL beaker	mL	
5.2	Warm the solution and maintain the temperature at 60°C by keeping the beaker on a heating mantle .		
5.3	Add KMnO ₄ from a burette with continuous stirring till pale pink color appears.		
5.4	Titre value for Standardization is	mL	
6.	Procedure:		
6.1	Transfer 100 mL, 50 mL and 50 mL of potassium permanganate solution, with a burette into 1 st , 2 nd and 3 rd absorption bottles respectively.		
6.2	Take 100 mL of sulphuric acid (10 % aqueous) solution in the reaction flask. (Sufficient quantity to see that the 'T' joint tube dips in H ₂ SO ₄).		
6.3	Assemble the apparatus pass the N ₂ gas at least for 30 minutes at 2-3 bubbles/sec so as to displace the air and make the atmosphere inert. (Tighten all the joints between the absorption bottles and make the assembly leak proof).		
6.4	Note down the percentage active ingredient declared on the sample	%	
6.5	Weigh accurately 0.3 g of sample on an ordinary filter paper.	g	
6.6	<i>Note down the S.No. of balance log book.</i>		
6.7	Transfer the sample along with filter paper to the reaction flask at once by opening the lid of the reaction flask slightly and close the lid immediately.		
6.8	Pass the Nitrogen gas and Keep for reaction for 30 min. at room temperature.		

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Prepared By		Checked By	Approved By
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6.9	During the process of setting the assembly, immerse the reaction flask in the water bath.		
6.10.	After 30 min, increase the temperature of water bath and maintain the temperature of the water bath at 65 ± 5 °C. Continue the reaction at least for 1 hr. at this temperature.		
6.11.	Sweep the last traces of phosphine from the flask with more rapid stream of nitrogen for 5 minutes.		
6.12.	At the end of the reaction disconnect the apparatus and quantitatively transfer the reduced potassium permanganate solution in the three absorption bottles to a 1000 mL beaker.		
6.13.	Rinse the absorption bottles and connecting tubes by adding 200 ml of 0.5 N oxalic acid solution through burette and add the rinsing to the reduced KMnO_4 solution.		
6.14.	Warm the contents of the beaker to approximately 60°C and titrate the excess oxalic acid at this temperature with standard 0.5N KMnO_4 solution, taken in a burette till pale pink colour end point appears.		
6.15	Titre value for sample is	mL	

7. Calculation:

$$1. \text{ Normality of Oxalic acid} = \frac{\text{Actual weight of oxalic acid taken} \times 0.5}{\text{Eq. wt. of Oxalic acid (15.75)}}$$

$$2. \text{ Normality of } \text{KMnO}_4 : V_1 N_1 = V_2 N_2$$

Where,

V_1 = Volume of standard potassium permanganate solution consumed.

V_2 = Volume of standard Oxalic acid solution pipette out

N_1 = Normality of standard potassium permanganate solution

N_2 = Normality of standard Oxalic acid solution

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$$\text{Aluminium Phosphide content \% by mass} = \frac{0.7247 [(200 + A)N_1 - 200 N_2]}{M}$$

Where,

A = Volume in ml of standard potassium permanganate solution required for the titration of excess Oxalic acid

N₁ = Normality of standard potassium permanganate solution

N₂ = Normality of standard Oxalic acid solution

M = Mass in g of the material taken for the test.

8. Result:

S.No.	Name of the Test	Result	Unit	Method of Analysis
1.	Active ingredient		%	IS:64351-1980 (Reaffirmed 2009)
Reference/ Remarks:				

Analyzed by	Name	
	Dated signature	
Checked by	Name	
	Dated signature	

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