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

Sr. No. in Scope NABL / NON NABL

Flow chart for analysis of Zinc 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 soloution :**		
3.1	Weigh 15.9 g of Potassium permanganate ($KMnO_4$) in a 250 mL beaker.	g	
3.2	*Note down the S.No. of balance log book.*		
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 15.8 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	*Note down the S.No. of balance log book.*		
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 $KMnO_4$ 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	Measure 100 mL of 1 N standard sodium hydroxide solution into the first glass absorption bottle.		
6.2	Transfer 100 mL, 50 mL and 50 mL of potassium permanganate solution, with a burette into 2nd , 3rd and 4th absorption bottles respectively.		
6.3	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_2SO_4).		
6.4	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).		
6.5	Note down the percentage active ingredient declared on the sample	%	
6.6	Weigh accurately 0.5 g of sample on an ordinary filter paper.	g	
6.7	*Note down the S.No. of balance log book.*		
6.8	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.		

Name of the Laboratory :	**Pesticide Formulation & Residue Analytical Centre, PMD, NIPHM, Hyderabad**				
Document No.	:	FC-PF-230	Document Name	:	Flow chart for analysis of Zinc phosphide in Formulation.
Revision No.	:	00	Issue Date	:	01/06/2013
Revision Date	:		Next Revision Date	:	01/06/2015

Prepared By	Checked By	Approved & Issued By
Mrs. C. Vijaya Lakshmi (Assistant Plant Protection Officer)	Mr. C.V. Rao (Technical Manager)	Dr. Abhay Ekbote (Director PM & Quality Manager)

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

6.9	Pass the Nitrogen gas and Keep for reaction for 30 min. at room temperature.		
6.10	During the process of setting the assembly, immerse the reaction flask in the water bath.		
6.11.	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.		
6.12.	Sweep the last traces of phosphine from the flask with more rapid stream of nitrogen for 5 minutes.		
6.13.	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.		
6.14.	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.		
6.15.	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.		
6.16	Titre value for sample is	mL	

7. Calculation:

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

2. Normality of $KMnO_4$: $V_1 N_1 = V_2 N_2$

Where,

V_1 = Volume of standard potassium permanganate solution consumed.

V_2 = Volume of standard Oxalic acid solution pipette out

N_1 = Noramality of standard potassium permanganate solution

N_2 = Normality of standard Oxalic acid solution

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

3. Zinc Phosphide content % by mass = $\frac{1.613\,[(200 + A)N_1 - 200\,N_2]}{M}$

Where,

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

N_1 = Noramality of standard potassium permanganate solution

N_2 = 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:1251-1988 (Reaffirmed 2009)
Reference/ Remarks:				

Analyzed by	Name	
	Dated signature	
Checked by	Name	
	Dated signature	

Name of the Laboratory :	**Pesticide Formulation & Residue Analytical Centre, PMD, NIPHM, Hyderabad**				
Document No.	:	FC-PF-230	Document Name	:	Flow chart for analysis of Zinc phosphide in Formulation.
Revision No.	:	00	Issue Date	:	01/06/2013
Revision Date	:		Next Revision Date	:	01/06/2015

Prepared By	Checked By	Approved & Issued By
Mrs. C. Vijaya Lakshmi (Assistant Plant Protection Officer)	Mr. C.V. Rao (Technical Manager)	Dr. Abhay Ekbote (Director PM & Quality Manager)