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

Sr. No. in Scope NABL/ NON NABL

Flow chart for analysis of Suspensibility of Deltamethrin WP formulation

Date of Analysis	

Sl. No.	Step	Execution		Executed By
1.	Sample No.			
2.	Name of Sample			
3.	**Procedure**	**R_1**	**R_2**	
3.1.	**Preparation of Standard**			
3.1.1	Purity of standard	%	%	
3.1.2	Weigh 0.05 g a.i. of standard in a 50 ml volumetric flask	g	g	
3.1.3	*Note down the S.No. of balance log book.*			
3.1.4	Dissolve and dilute up to the mark with a mixture of 1,4-dioxan and iso-octane (20 : 80)			
3.1.5	Pipette out 2 mL of standard solution (3.2.4) into a 10 mL volumetric f lask and make up to the mark with a mixture of 1,4-dioxan and iso-octane (20 : 80)			
3.2	**Preparation of suspension:**			
3.2.1	Weigh 0.05 g a. i. sample in a 100 ml beaker			
3.2.2	*Note the serial No. of balance log book*			
3.2.3	Add standard hard water 342 ppm (at least twice the mass of the material taken for test) at 30 ± 1^0C			
3.2.4	Allow to stand for 30 sec. & stir by hand for 30 sec. with a glass rod.			
3.2.5	Transfer the slurry to the stoppered measuring cylinder (250 ml) and any residue by washing with small quantity of hard water (342 ppm)			
3.2.6	Add hard water (342 ppm) up to the mark			
3.2.7	Close the cylinder with the stopper and invert it sharply through 30 complete cycles within 1 min.			
3.2.8	Allow the cylinder to stand at rest for 30 min. at 30±1^0C			
3.2.9	Withdraw suspension (nine-tenths) from the cylinder within 10 to 15 sec by dipping the nozzle of the glass tube using suction through filtration flask			
3.2.10	Suspension including sediment at the bottom of the cylinder (one - tenth of the suspension)			

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

3.3	**Preparation of Sample**			
3.3.1	Transfer the bottom 25 mL of sediment to a 250 mL separating funnel.			
3.3.2	Extract the sample with mixture iso-octane : 1,4-dioxane(80 :20) thrice using 25 mL each time.			
3.3.3	Collect the organic solvent layer in a 500 mL GG stoppered conical flask passing through a funnel plugged with cotton and filled anhydrous Na_2SO_4			
3.3.4	Evaporate the solvent on a rotary vaccum evaporator at 85°C to less than 5 mL			
3.3.5	Quantitatively transfer the content to a 50 mL volumetric flask using a mixture of 1,4-dioxan and iso-octane (20 : 80) and make up to the mark.			
3.3.6	Filter the sample through sample filtration kit using 0.45µm membrane filters and inject the sample.			
4.	**HPLC Parameters**			
4.1	**Column**			
4.1.1	S.S. Packed with Lichrosorb silica 60-80 mesh			
4.1.2	Length: 25 cm			
4.1.3	I.D.: 4.6 mm			
4.2	**Mobile Phase**			
4.2.1	Dioxan: iso-octane (5 : 95)			
4.2.2	Flow Rate: 1.5 ml/min			
4.3	**Detector:** Ultra-violet			
4.4	**Wave Length**: 254 nm			
4.5	**Injection Volume:** 20 µl			
5.	**Result**			
	Sample chromatogram no.			
	Standard chromatogram no.			

6. Calculations:

Deltamethrin content in (g), in bottom 25 mL of sediment is = $= \frac{A_1 \times M_1 \times P}{A_2 \times 5 \times 100}$	Where, A_1 = Area of Deltamethrin in sample solution A_2 = Area of Deltamethrin in standard solution M_1 = Weight of Deltamethrin standard taken in g. P = Purity of Deltamethrin standard

Name of the Laboratory :		**Pesticide Formulation & Residue Analytical Centre, PMD, NIPHM, Hyderabad**			
Document No.	:	FC-PF-277	Document Name	:	Flow chart for analysis of suspensibility of Deltamethrin WP formulation, % by mass
Revision No.	:	00	Issue Date	:	01/06/2014
Revision Date	:	--	Next Revision Date	:	01/06/2016

Prepared By	Checked By	Approved By	Issued By
Mrs.T. Sridevi (Deputy Quality Manager)	Dr. Nirmali Saikia (Technical Manager)	Dr. Abhay Ekbote (Director PM)	Mr. C. V. Rao (Quality Manager)

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

$$\% \text{ Suspensibility} = \frac{1000 \text{ X } (M-m)}{9 \text{ X } M}$$

Where,

M = Mass in 'g' of pesticide present in the sample taken for the preparation of suspension

m = Mass in 'g' of pesticide found in the suspension 1/10th of sediment remaining in the graduated cylinder

Result:

Sl. No.	Name of test	Result	Unit	Method of Analysis
1.	Suspensibility		%	IS: 6940-1982
Remark / Reference :				

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-277	Document Name	:	Flow chart for analysis of suspensibility of Deltamethrin WP formulation, % by mass
Revision No.	:	00	Issue Date	:	01/06/2014
Revision Date	:	--	Next Revision Date	:	01/06/2016

Prepared By	Checked By	Approved By	Issued By
Mrs.T. Sridevi (Deputy Quality Manager)	Dr. Nirmali Saikia (Technical Manager)	Dr. Abhay Ekbote (Director PM)	Mr. C. V. Rao (Quality Manager)