

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

Sr. No. in Scope

NABL / NON NABL

Flow Chart of Analysis of Suspensibility test in Lambda-Cyhalothrin Wettable Powder (WP) formulation

| | | Date of Analysis | |
|---------|--|------------------|-------------|
| Sr. No. | Step | Execution | Executed By |
| 1. | Sample No. | | |
| 2.1 | Name of Sample | | |
| 2.2 | Sample Description | | |
| 3. | Procedure | | |
| 3.1 | Preparation of suspension | | |
| 3.1.1 | Weigh sample equivalent to 50 mg of a.i. in 100 ml beaker | g | |
| 3.1.2 | Add standard hard water 342 ppm (at least twice the mass of the material taken for test) at $30 \pm 1^{\circ}\text{C}$ | | |
| 3.1.3 | Allow to stand for 30 sec. & stir by hand for 30 sec. with a glass rod | | |
| 3.1.4 | Transfer the slurry to the graduated cylinder (250 ml) and any residue by washing with small quantity of hard water (342 ppm) | | |
| 3.1.5 | Add hard water (342 ppm) up to the mark | | |
| 3.1.6 | Close the cylinder with the stopper and invert it sharply through 30 complete cycles within 1 min. | | |
| 3.1.7 | Allow the cylinder to stand at rest for 30 min. at $30 \pm 1^{\circ}\text{C}$ | | |
| 3.1.8 | 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 | | |
| 3.1.9 | Suspension including sediment at the bottom of the cylinder (one - tenth of the suspension) | mL | |
| 3.2 | Preparation of Standard | | |
| 3.2.1 | Weigh 50mg Standard in 50mL volumetric flask | | |
| 3.2.2 | Purity of standard | | |
| 3.2.3 | Dilute up to the mark with hexane | | |
| 3.2.4 | Take 10 mL of the solution (3.2.3) to a 50 ml volumetric flask | | |
| 3.2.5 | Dilute up to the mark with hexane | | |

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| Name of the Laboratory : Pesticide Formulation & Residue Analytical Centre, PMD, NIPHM, Hyderabad | | | | | |
| Document No. | : | FC-PF-239 | Document Name | : | Flow chart for analysis of Suspensibility test in Lambda - Cyhalothrin WP |
| Revision No. | : | 00 | Issue Date | : | 01/07/2011 |
| Revision Date | : | | Next Revision Date | : | 01/07/2013 |
| Prepared By | | Checked By | | Approved & Issued By | |
| Mrs. T. Sridevi (Deputy Technical Manager) | | Mr. C.V. Rao (Technical Manager) | | Dr Abhay Ekbote (Director – PM & Quality Manager) | |

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|-------------|---|--|--|
| 3.3 | Determination of Active Ingredient | | |
| 3.3.1 | Transfer 1/10 th of the bottom suspension quantitatively into 250 mL separating funnel. | | |
| 3.3.2 | Use 25mL water to rinse the cylinder. Combine suspension and washings. | | |
| 3.3.3 | Add 25mL of Dichloromethane to the separating Funnel. Shake for one min. and leave for separation. | | |
| 3.3.4 | Add saturated NaCl solution if emulsion is formed. | | |
| 3.3.5 | Transfer Dichloromethane layer into a 250ml round bottom flask. | | |
| 3.3.6 | Repeat the extraction three more times each time using 25mL Dichloromethane. | | |
| 3.3.7 | Combine all Dichloromethane extract in to round bottom flask (GG neck). | | |
| 3.3.8 | Remove Dichloromethane under rotary vacuum evaporator at 60 °C. | | |
| 3.3.9 | Dissolve the residue in chloroform transfer into 25mL volumetric flask and make up with chloroform. | | |
| 4. | HPLC Parameters | | |
| 4.1 | 4.1 Column | | |
| 4.1.1 | Stainless Steel packed with Partisil Silica 5 µ | | |
| 4.1.2 | Length: 25 cm | | |
| 4.1.3 | I.D.: 4.6 mm | | |
| 4.2 | Mobile Phase | | |
| 4.2.1 | Hexane : Tetrahydrofuran (99.5 : 0.5) | | |
| 4.2.2 | Flow Rate: 1.5 ml/min | | |
| 4.3 | Detector: UV | | |
| 4.4. | Wave Length: 235 nm | | |
| 4.5. | Injection Volume: 20 µl | | |
| 5. | Result | | |
| | Sample chromatogram no. | | |
| | Standard chromatogram no. | | |

6. Calculation:

| | | | | | |
|--|---|-------------------------------------|--------------------|--|---|
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|--|---|
| <p>Lambda-Cyhalothrin $A_2 \times M_1$ content, % by mass = $\frac{\text{-----}}{A_1 \times 100} \times P \times \text{Dilution Factor}$</p> | <p>$M_1$ = Mass in 'g' of Lambda- Cyhalothrin standard M_2 = Mass in 'g' of sample taken for test A_1 = Peak area of Lambda- Cyhalothrin in the standard solution A_2 = Peak area of Lambda- Cyhalothrin in the sample solution P = Percent purity of Lambda- Cyhalothrin in the standard Dilution Factor = 1/5</p> |
| <p>Suspensibility, $\% \text{ by mass} = \frac{1000 (M - m)}{9 M}$</p> | <p>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</p> |

| Sr. 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 | |

| | | | | | |
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