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 ± 10C | |  |  |
| 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 ± 10C | |  |  |
| 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 | |  |  |

|  |  |  |  |
| --- | --- | --- | --- |
| **3.3** | **Determination of Active Ingredient** |  |  |
| 3.3.1 | Transfer 1/10th 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:** 20µl |  |  |
| **5.** | **Result** |  |  |
|  | Sample chromatogram no. |  |  |
|  | Standard chromatogram no. |  |  |

**6. Calculation:**

|  |  |
| --- | --- |
| Lambda-Cyhalothrin A2 x M1  content, % by mass = ------------- x P X Dilution Factor  A1 x 100 | M1 =Mass in ‘g’ of Lambda- Cyhalothrin standard  M2 =Mass in ‘g’ of sample taken for test  A1 =Peak area of Lambda- Cyhalothrin   in the standard solution  A2 = Peak area of Lambda- Cyhalothrin   in the sample solution  P = Percent purity of Lambda-Cyhalothrin in the standard  Dilution Factor = 1/5 |
| **Suspensibility,**  1000 (M - m)  **% by mass** = -----------------------  9 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 |

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Sr. No.** | **Name of test** | **Result** | **Unit** | **Method of Analysis** |
| 1. | Suspensibiltiy |  | % | IS : 6940 - 1982 |
| Remark / Reference : | | | | |

|  |  |  |
| --- | --- | --- |
| Analyzed by | Name |  |
| Dated signature |  |
| Checked by | Name |  |
| Dated signature |  |