Sr. No. in Scope NABL / NON NABL

**Flow chart for analysis of Acetamiprid content in formulation sample**

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| --- | --- |
| **Date of Analysis**  |  |

|  |  |  |  |
| --- | --- | --- | --- |
| **S. No.** | **Step**  | **Execution** | **Executed by**  |
| 1. | Sample No. | **R1** | **R2** |  |
| 2. | Name of Sample |  |  |  |
| 3. | **Procedure** |  |  |  |
| **3.1.** | **Preparation of Mobile Phase**  |  |  |  |
| 3.1.1. | Mix acetonitrile , water and 10% Ortho phosphoric acid in the proportion of 60:38:2 (v/v) |  |  |  |
| 3.1.2. | Pass through the membrane filter under vaccum. |  |  |  |
| 3.1.3. | Homogenize the mixture using the magnetic stirrer. |  |  |  |
| 3.1.4. | Allow to attain room temperature. |  |  |  |
| **3.2** | **Preparation of Internal Standard Solution** |  |  |  |
| 3.2.1 | Weigh 0.45g of Acetophenone in 100 ml volumetric flask | g | g |  |
| 3.2.2 | *Note the serial No. of the balance log book* |  |  |  |
| 3.2.3 | Dissolve and dilute up to the mark with acetonitrile |  |  |  |
| **3.3** | **Preparation of standard solution** |  |  |  |
| 3.3.1 | Note the purity of the standard | % | % |  |
| 3.3.2 | Weigh 0.025 g a.i. of Standard in a 50 ml volumetric flask  | g | g |  |
| 3.3.3 | *Note the serial No. of the balance log book* |  |  |  |
| 3.3.4 | Add 5ml of internal standard solution (3.2.3) | mL | mL |  |
| 3.3.5 | Dilute up to the mark with acetonitrile (Stock A) |  |  |  |
| 3.3.6 | Pipette out 2 mL of Stock A (3.3.5) into a 25 mL volumetric flask |  |  |  |
| 3.3.7 | Dilute up to the mark with acetonitrile |  |  |  |
| **3.4** |  **Preparation of sample solution** |  |  |  |
| 3.4.1 | Note the percent active ingredient content declared on sample | % | % |  |
| 3.4.2 | Weigh 0.025 g a.i. of the sample in 50 ml volumetric flask | g | g |  |
| 3.4.3 | *Note the serial No. of the balance log book* |  |  |  |
| 3.4.4 | Add 5ml of internal standard solution (3.2.3) | ml | ml |  |
| 3.4.5 | Dilute up to the mark with acetonotrile (Stock B) |  |  |  |
| 3.4.6 | Pipette out 2ml of Stock B (3.4.5) into a 25ml volumetric flask |  |  |  |
| 3.4.7 | Dilute up to the mark with acetonitrile |  |  |  |
| 3.4.8 | Filter the sample solution through 0.45µ membrane filter |  |  |  |
| 4. | **HPLC Parameters** |  |  |  |
| **4.1** | **Column** |  |  |  |
| 4.1.1 | C18, Particle Size: 5µ  |  |  |  |
| 4.1.2 | Length: 250 mm |  |  |  |
| 4.1.3 | I.D.: 4.6 mm |  |  |  |
| **4.2** | **Mobile Phase** |  |  |  |
| 4.2.1 | Acetonitrile : Water : 10% Phosphoric acid (60:38:2) |  |  |  |
| 4.2.2 | Flow Rate : 1 ml/min |  |  |  |
| **4.3** |  **Detector:** UV |  |  |  |
| **4.4** |  **Wave Length**: 250 nm |  |  |  |
| **4.5** |  **Injection Volume:** 20µl |  |  |  |
| 5. | **Result** |  |  |  |
| Sample chromatogram no.  |  |  |  |
| Standard chromatogram no.  |  |  |  |

**6. Calculation:**

|  |  |
| --- | --- |
|  A2 x A3 x M1 Acetamiprid content, = ------------------- x P% by mass A1 x A4 x M2 | **Where,**M1 =Mass in ‘g’ of acetamiprid standardM2 =Mass in ‘g’ of sample taken for test A1 = Peak area of acetamiprid in the standard solutionA2 = Peak area of acetamiprid in the sample solutionA3 = Peak area of internal standard in the standard solution A4 = Peak area of internal standard in the sample solution P = Percent purity of acetamiprid standard |

**Calculation for R1**

**Calculation for R2**

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Sr. No.** | **Name of test** | **Result** | **Unit** | **Method of Analysis**  |
| 1. | Active ingredient |  | % | CIBRC approved method |
| Remark / Reference : |

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