Sr. No. in Scope NABL / NON NABL

**Flow Chart for Analysis of Thiamethoxam Content in Formulation Sample**

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

|  |  |  |  |
| --- | --- | --- | --- |
| **S. No.** | **Step**  | **Execution** | **Executed by**  |
| 1. | Sample No. |  |  |  |
| 2. | Name of Sample |  |  |  |
| 3. | Sample Description |  |  |  |
| **4.** | **Procedure** | R1 | R2 |  |
| **4.1.** | **Preparation of Mobile Phase**  |  |  |  |
| 4.1.1 | First prepare 1% Ortho phosphoric acid in water and pass through the membrane filter under vacuum. |  |  |  |
| 4.1.2 | Mix Methanol and 1% Ortho phosphoric acid in water (4.1.1) in the proportion of 25:75 (v/v). |  |  |  |
| 4.1.3 | Homogenize the mixture and keep for sonication under ultrasonic bath. |  |  |  |
| 4.1.4 | Allow to attain room temperature. |  |  |  |
| **4.2** | **Preparation of Standard solution** |  |  |  |
| 4.2.1 | Note the purity of the standard | % | % |  |
| 4.2.2 | Weigh 50 mg a.i. of Standard into a 100 ml volumetric flask  | mg | mg |  |
| 4.2.3 | *Note the serial No. of the balance log book* |  |  |  |
| 4.2.4 | Dissolve and dilute up to the mark with Mobile phase (4.1.4) [Stock A] |  |  |  |
| 4.2.5 | Pipette out 5 mL of Stock A (4.2.4) into a 10 mL volumetric flask | ml | ml |  |
| 4.2.6 | Dilute up to the mark with Mobile phase (4.1.4). |  |  |  |
| **4.3** | **Preparation of Sample solution** |  |  |  |
| 4.3.1 | Note the percent active ingredient content declared on sample | % | % |  |
| 4.3.2 | Weigh accurately a quantity of Sample to contain 50 mg a. i. into a 100 ml volumetric flask  | mg | mg |  |
| 4.3.3 | *Note the serial No. of the balance log book* |  |  |  |
| 4.3.4 | Dissolve and dilute up to the mark with Mobile phase (4.1.4) [Stock B] |  |  |  |
| 4.3.5 | Pipette out 5 mL of Stock B (4.3.4) into a 10 mL volumetric flask | ml |  |  |
| 4.3.6 | Dilute up to the mark with Mobile phase (4.1.4). |  |  |  |
| 4.3.7 | Filter the sample solution through 0.45µ membrane filter |  |  |  |
| **5.** | **HPLC Parameters** |  |  |  |
| **5.1** | **Column** |  |  |  |
| 5.1.1 | C18, Particle Size: 5µ  |  |  |  |
| 5.1.2 | Length: 250 mm |  |  |  |
| 5.1.3 | I.D.: 4.6 mm |  |  |  |
| **5.2** | **Mobile Phase** |  |  |  |
| 5.2.1 | Methanol and 1% Ortho phosphoric acid in water (25:75) |  |  |  |
| 5.2.2 | Flow Rate : 1 ml/min |  |  |  |
| **5.3** |  **Detector :** UV  |  |  |  |
| **5.4** |  **Wave Length** : 254 nm |  |  |  |
| **5.5** |  **Injection Volume :** 20µl |  |  |  |
| **6.** | **Result** |  |  |
| Sample chromatogram no.  |  |  |
| Standard chromatogram no.  |  |  |

**7. Calculation:**

|  |  |
| --- | --- |
|  A2 x M1 Thiamethoxam content, = -------------- x P% by mass A1 x M2 | **Where,**M1 =Mass in ‘mg’ of Thiamethoxam standard M2 =Mass in ‘mg’ of sample taken for test  A1 = Peak area of Thiamethoxam in the standard solution A2 = Peak area of Thiamethoxam in the sample solution P = Percent purity of Thiamethoxam standard |

**Result:**

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **SI. No.** | **Name of test** | **Result** | **Unit** | **Method of Analysis** |
| 1. | Active ingredient |  | % | In house method |
| Remark / Reference : |
|  |
| Analyzed by | Name  |  |
| Dated signature |  |
| Checked by | Name  |  |
| Dated signature |  |