Sr. No. in Scope NABL / NON NABL

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

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

|  |  |  |  |
| --- | --- | --- | --- |
| **Sl. No.** | **Step** | **Execution** | **Executed By** |
| 1. | Sample No. |  |  |
| 2. | Name of Sample | | |
| 3. | **Procedure** | | |
| **3.1** | **Preparation of Mobile Phase** |  |  |
| 3.1.1 | Mix acetonitrile and water in the proportion of 80:20 (v/v) |  |  |
| 3.1.2 | Pass through membrane filter under vacuum |  |  |
| 3.1.3 | Homogenize the mixture using a magnetic stirrer |  |  |
| 3.1.4 | Allow to attain room temperature |  |  |
| **3.2** | **Preparation of Internal Standard Solution** |  |  |
| 3.2.1 | Weigh 0.03g of Acenaphthene in 100 ml volumetric flask. | g |  |
| 3.2.2 | *Note the serial No. of the balance log book* |  |  |
| 3.2.3 | Add to it 40 ml of mobile phase (3.1.4) |  |  |
| 3.2.4 | Keep it for 30 minute with intermittent shaking |  |  |
| 3.2.5 | Dilute up to the mark with mobile phase (3.1.4) |  |  |
| **3.3** | **Preparation of Standard Solution** |  |  |
| 3.3.1 | Note the purity of the standard | % |  |
| 3.3.2 | Weigh 0.1g A.I. of standard in 100 ml volumetric flask | g |  |
| 3.3.3 | *Note the serial No. of the balance log book* |  |  |
| 3.3.4 | Add to it 40 ml of mobile phase (3.1.4) |  |  |
| 3.3.5 | Keep it for 30 minute with intermittent shaking |  |  |
| 3.3.6 | Dilute up to the mark with mobile phase (3.1.4) |  |  |
| 3.3.7 | Pipette out 10ml of solution (3.3.6) to a 100 ml volumetric flask | ml |  |
| 3.3.8 | Dilute up to the mark with mobile phase (3.1.4) |  |  |
| 3.3.9 | Pipette out 20ml of solution (3.3.8) to a 100 ml volumetric flask | ml |  |
| 3.3.10 | Add 20ml of internal standard solution (3.2.5) | ml |  |
| 3.3.11 | Dilute up to the mark with mobile phase (3.1.4) |  |  |
| **3.4** | **Preparation of Sample** |  |  |
| 3.4.1 | Note the percent active ingredient content declared on sample | % |  |
| 3.4.2 | Weigh 0.1g A.I. of sample in 100 ml volumetric flask | g |  |
| 3.4.3 | *Note the serial No. of the balance log book* |  |  |
| 3.4.4 | Add to it 40 ml of mobile phase (3.1.4) |  |  |
| 3.4.5 | Keep it for 30 minute with intermittent shaking |  |  |
| 3.4.6 | Dilute up to the mark with mobile phase (3.1.4) |  |  |
| 3.4.7 | Pipette out 10ml of solution (3.4.6) to a 100 ml volumetric flask | ml |  |
| 3.4.8 | Dilute up to the mark with mobile phase (3.1.4) |  |  |
| 3.4.9 | Pipette out 20ml of solution (3.4.8) to a 100 ml volumetric flask | ml |  |
| 3.4.10 | Add 20ml of internal standard solution (3.2.5) | ml |  |
| 3.4.11 | Dilute up to the mark with mobile phase (3.1.4) |  |  |
| **4.** | **HPLC Parameters** |  |  |
| **4.1** | **Column** |  |  |
| 4.1.1 | Stainless Steel Packed with Novapak C18 |  |  |
| 4.1.2 | Length: 250 mm |  |  |
| 4.1.3 | I.D.: 4.6 mm |  |  |
| **4.2** | **Mobile Phase** |  |  |
| 4.2.1 | Acetonitrile : Water (80 : 20) |  |  |
| 4.2.2 | Flow Rate: 1.0 ml/min |  |  |
| **4.3** | **Detector:** UV |  |  |
| **4.4** | **Wave Length**: 278 nm |  |  |
| **4.5** | **Injection Volume:** 20 µl |  |  |
| **5.** | **Result** |  |  |
| Sample chromatogram no. |  | |
| Standard chromatogram no. |  | |

**6. Calculation:**

A2 x A3 x M1

Imidacloprid content, % by mass = ------------------- x P

A1 x A4 x M2

**Where,**

M1 = Mass in ‘g’ of imidacloprid standard

M2 =Mass in ‘g’ of sample taken for test

A1 = Peak area of imidacloprid in the standard solution

A2 = Peak area of imidacloprid in the sample solution

A3 = Peak area of internal standard in the standard solution

A4 = Peak area of internal standard in the sample solution

P = Percent purity of imidacloprid in the standard

**Result:**

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Sl. No.** | **Name of test** | **Result** | **Unit** | **Method of Analysis** |
| 1. | Active ingredient |  | % | 1S 15443 : 2004  (Reaffirmed 2009) |
| Remark / Reference : | | | | |

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