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| **Sr. No. in Scope** | **NABL / NON NABL** |

**Flow chart for analysis of Fenvalerate in Dusting Powder formulation sample**

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| --- | --- | --- | --- |
| **Date of Analysis** | |  | |
| **S.No.** | **Step** | **Execution** | | | | **Executed**  **By** |
| **R1** | | **R2** | |
| 1. | Sample No. |  | |  | |  |
| 2. | Name of Sample |  | |  | |  |
| 3. | **Procedure** |  | |  | |  |
| **3.1** | **Preparation of Internal Standard** |  | |  | |  |
| 3.1.1 | Weigh 0.6 g of Di (2-ethylhexyl) Phthalate (DOP) in a 100 mL volumetric flask. | g | | g | |  |
| 3.1.2 | *Note down the S.No. of balance log book* |  | |  | |  |
| 3.1.3 | Dissolve and dilute up to the mark with chloroform |  | |  | |  |
| **3.2** | **Preparation of Standard Solution** |  | |  | |  |
| 3.2.1 | Purity of standard | % | | % | |  |
| 3.2.2 | Weight 0.080 g a.i. of standard in a 50 mL volumetric flask | g | | g | |  |
| 3.2.3 | *Note down the S.No. of balance log book* |  | |  | |  |
| 3.2.4 | Add 10 mL of internal standard solution (3.1.3) | mL | | mL | |  |
| 3.2.5 | Make up to the mark with chloroform |  | |  | |  |
| **3.3** | **Preparation of Sample Solution** |  | |  | |  |
| 3.3.1 | Note down the percent active ingredient content declared on the sample | % | | % | |  |
| 3.3.2 | Weigh sample so as to contain 0.080 g a.i. (20 g of DP) in a 250 mL beaker | g | | g | |  |
| 3.3.3 | *Note down the S.No. of balance log book* |  | |  | |  |
| 3.3.4 | Add 40 mL of acetone, stir intermittently and set it aside for 30 minutes. |  | |  | |  |
| 3.3.5 | Filter the sample under suction through G4 sintered funnel. |  | |  | |  |
| 3.3.6 | Repeat the extraction from the residue 5 times using 10 mL of acetone each time. |  | |  | |  |
| 3.3.7 | Collect the filtrate into a 250 mL filteration flask, and evopate the sample on a water bath to near dryness. |  | |  | |  |
| 3.3.8 | Transfer the sample quantitatively to a 50 mL volumetric flask using 25-30 mL of chloroform. |  | |  | |  |
| 3.3.9 | Add 10 mL of internal standard solution (3.1.3) |  | |  | |  |
| 3.3.10 | Make up to the mark with chloroform. |  | |  | |  |
| 4. | **GC Parameters** |  | |  | |  |
| **4.1** | **Column :** SS packed with 5 % OV 101 on chromosorb WHP (80-100) mesh |  | |  | |  |
| 4.1.1 | Length: 50 cm |  | |  | |  |
| 4.1.2 | I.D: 0.3 mm |  | |  | |  |
| **4.2** | **Gas Flow:** |  | |  | |  |
| 4.2.1 | Carrier:Nitrogen: 30 mL/min |  | |  | |  |
| 4.2.2 | Hydrogen: 45 mL/min |  | |  | |  |
| 4.2.3 | Air: 450 mL/min |  | |  | |  |
| **4.3** | **Temperatures** |  | |  | |  |
| 4.3.1 | Oven: 2400C |  | |  | |  |
| 4.3.2 | Injecter: 2600C |  | |  | |  |
| 4.3.3 | Detector: 2800C |  | |  | |  |
| **4.4** | **Injection Volume:** 1 µl |  | |  | |  |
| 5. | **Data:** |  | |  | |  |
| Sample chromatogram no |  | |  | |  |
| Standard chromatogram no |  | |  | |  |

**6. CALCULATION:**

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| --- | --- |
| Fenvalerate content, % by mass =    A1 x A’IS’2 x M1  ------------------------ X P  A’IS’1 x A2 x M2 | **Where,**  A1 = Peak area of fenvalerate in the sample solution  A’IS’1 = Peak area of internal standard in the sample  A’IS’2 = Peak area of internal standard in the standard solution  A2 = Peak area of fenvalerate in the standard solution  M1 = Mass in ‘g’ of standard fenvalerate in the standard solution M2 = Mass in ‘g’ of fenvalerate sample taken for test  P = Percent purity of fenvalerate standard |

**Result:**

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **S1 No.** | **Name of test** | | **Result** | | **Unit** | **Method of Analysis** |
| 1. | Active ingredient | |  | | % | IS: 12003 – 1987 (Tech.)& IS- 15341-2003(DP) |
| Remark / Reference: | | | | | | |
| Analyzed by | | Name | |  | | |
| Dated signature | |  | | |
| Checked by | | Name | |  | | |
| Dated signature | |  | | |