Sr. No. in Scope NABL / NON NABL

 **Flow chart for analysis of Chlorpyrifos and Cypermethrin in formulation sample**

|  |  |
| --- | --- |
| **Date of Analysis**  |  |
| **Sl. No.** | **Step**  |  **Execution** | **Executed**  **By**  |
| 1. | Sample No. |  |  |
| 2. | Name of Sample |  |  |
| 3. | **Procedure** |
| **3.1** | **Preparation of Internal Standard**  |  |  |
| 3.1.1 | Weigh 0.5 g Dioctyl phthalate (DOP) taken into 250 ml volumetric flask |  g |  |
| 3.1.2 | *Note down the S.No. of balance log book.* |  |  |
| 3.1.3 | Dissolve it and make the volume up to the mark with ethyl acetate |  |  |
| **3.2** | **Preparation of Standard Cypermethrin Solution** |  |  |
| 3.2.1 | Purity of standard Cypermethrin |  % |  |
| 3.2.2 | Weigh 0.15 g a.i of standard into a 50 ml volumetric flask. |  g |  |
| 3.2.3 | *Note down the S.No. of balance log book.* |  |  |
| 3.2.4 | Disolve and dilute up to the mark with ethyl acetate (Stock A) |  |  |
| **3.3.** | **Preparation of mixture of Chlorpyrifos and Cypermethrin Standard Solution:** |  |  |
| 3.3.1 | Purity of standard Chlorpyrifos |  % |  |
| 3.3.2 | Weigh 0.15 g a.i. of chlorpyrifos standard into a 50 ml volumetric flask  |  g |  |
| 3.3.3 | *Note down the S.No. of balance log book.* |  |  |
| 3.3.4 | Add 5 mL of stock A (3.2.4) and 25 mL of internal standard solution (3.1.3) |   |  |
| 3.3.5 | Dilute up to the mark with ethyl acetate and mix well. |   |  |
| **3.4** | **Preparation of Sample** |  |  |
| 3.4.1 | Note down the percent active ingredient content of chlorpyrifos and cypermethrin declared on the sample, respectively. |  % % |  |
| 3.4.2 | Weigh 0.3 g of the sample into a 50 ml volumetric flask |  g |  |
| 3.4.3 | *Note down the S.No. of balance log book.*  |  |  |
| 3.4.4 | Add 25 mL of internal standard solution (3.1.3) |  ml |  |
| 3.4.5 | Dilute up to the mark with ethyl acetate and mix well. |   |  |
|  **4.** | **GC Parameters** |  |  |
|  **4.1** | **Column:** 3 % OV -101 on Gas chrom Q (80-100) mesh |  |  |
| 4.1.1 | Length: 100 cm |  |  |
| 4.1.2 | I.D: 2 mm |  |  |
|  **4.2** | **Gas** |  |  |
| 4.2.1 | Carrier:Nitrogen: 30 ml/min |  |  |
| 4.2.2 |  Hydrogen: 45 ml/min |  |  |
| 4.2.3 |  Air: 450 ml/min |  |  |
| **4.3** | **Temperature** |  |  |
| 4.3.1 | Oven: 1800C for 2.5 min @ 15°C/min upto 240°C for 2.5 min  |  |  |
| 4.3.2 | Injector: 2500C |  |  |
| 4.3.3 | Detector: 2700C |  |  |
| **4.4** | **Injection volume:** 1 µl |  |  |
|  5. | **Result**  |  |  |
|  | Sample chromatogram no.  |  |  |
| Standard chromatogram no.  |  |  |

**6. CALCULATION:**

|  |  |
| --- | --- |
|  **Chlorpyrifos content, % by mass =** A1 x A’IS’2 x M1 --------------------------- x P A’IS’1 x A2 x M2   | **Where,**A1= Peak area of chlorpyrifos in the sample solutionA’IS’1= Peak area of internal standard in the sample solutionA’IS’2= Peak area of internal standard in the standard solutionA2= Peak area of chlorpyrifos in the standard solutionM1= Mass in ‘g’ of chlorpyrifos in the standard solutionM2= Mass in ‘g’ of sample taken for test P = Percent purity of chlorpyrifos standard |

|  |  |
| --- | --- |
| **Cypermethrin content, % by mass =** A3 x A’IS’2 x M----------------------- x P A’IS’1 x A4 x M2   | **Where,**A3= Peak area of cypermethrin in the sample solutionA’IS’1= Peak area of internal standard in the sample solutionA’IS’2= Peak area of internal standard in the standard solutionA4= Peak area of cypermethrin in the standard solutionP = Percent purity of cypermethrin standardM2= Mass in ‘g’ of sample taken for test M= Mass in ‘g’ of cypermethrin in standard. |

 **Result:**

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Sl. No.** | **Name of Test** | **Result** | **Unit** | **Method of Analysis** |
| 1. | Active ingredient (Chlorpyrifos) |  | % |  (IS - 15235 : 2002) (Reaffirmed 2009) |
| 2. | Active ingredient (Cypermethrin) |  | % |
| Remark / Reference :  |

|  |  |  |
| --- | --- | --- |
| Analyzed by | Name  |  |
| Dated Signature |  |
| Checked by | Name  |  |
| Dated Signature |  |