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 solution  A’IS’1= Peak area of internal standard in the sample solution  A’IS’2= Peak area of internal standard in the standard solution  A2= Peak area of chlorpyrifos in the standard solution  M1= Mass in ‘g’ of chlorpyrifos in the standard solution  M2= 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 solution  A’IS’1= Peak area of internal standard in the sample solution  A’IS’2= Peak area of internal standard in the standard solution  A4= Peak area of cypermethrin in the standard solution  P = Percent purity of cypermethrin standard  M2= 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 |  |