Sr. No. in Scope NABL / NON NABL

**Flow Chart for Analysis of Prallethrin and Piperonyl Butoxide Content in**

**Mosquito Repellent Mats**

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

|  |  |  |  |
| --- | --- | --- | --- |
| **S. No.** | **Step**  | **Execution** | **Executed By**  |
| 1. | Sample No. |  |  |
| 2.1 | Name of Sample |
| 2.2 | Sample Description |
| 3. | **Procedure** |
| **3.1** | **Preparation of Internal Standard** |  |  |
| 3.1.1 | Weigh 1 g of Di-n-butyl Phthalate taken in a 100 mL volumetric Flask. |   |  |
| 3.1.2 | Make up the volume with Toluene. |  |  |
| **3.2**  | **Preparation of Reference Standard of Prallethrin** |  |  |
| 3.2.1 | Note the Purity of standard | % |  |
| 3.2.2 | Weigh standard equivalent to 1.0 g of active ingredient in 100 ml volumetric flask | g |  |
| 3.2.3 | Dilute up to the mark with Toluene. (Stock A)  |  |  |
| **3.3**  | **Preparation of Reference Standard of Piperonyl Butoxide**  |  |  |
| 3.3.1 | Note the Purity of standard |  |  |
| 3.3.2. | Weigh Standard equivalent to 1.0 g active ingredient in 100 mL volumetric flask. |  |  |
| 3.3.3 | Dilute up to the mark with Toluene. (Stock B) |  |  |
| **3.4** | **Preparation of Working Standard of Prallethrin and Piperonyl Butoxide** |  |  |
| 3.4.1. | Pipette out 4 mL Stock A solution of Prallethrin reference standard (3.2.3) and 1mL Stock B solution of Piperonyl butoxide reference standard (3.3.3) into an another 100 mL volumetric flask. |  |  |
| 3.4.2. | Add to it 4 mL of Internal Standard (3.1.2) solution. |  |  |
| 3.4.3 | Make up to the mark with Toluene. |  |  |
| 3.4.4. | Weight of Prallethrin Standard in working standard  |  |  |
| 3.4.5 | Weight of Piperonyl butoxide Standard in working standard  |  |  |
| **3.5.**  | **Extraction from Mosquito repellent mat and Preparation of sample solution** |  |  |
| 3.5.1 | Weigh one mat sample. | g |  |
| 3.4.2 | Peel and cut the mat into small pieces and transfer to a 250mL stoppered conical flask. |  |  |
| 3.4.3 | Add to it 60 ml of Toluene and shake on a mechanical shaker for 20 mins.  |  |  |
| 3.4.4 | Transfer the supernatant into a 100 mL volumetric flask.  |  |  |
| 3.4.5 |  Wash the conical flask and repeat the steps 3.4.3.and 3.4.4. two more time with 10 mL of toluene each time.  |  |  |
| 3.4.6 | Add to it 4 mL of Internal Standard (3.1.2.) |  |  |
| 3.4.7. | Make up to the mark with Toluene. |  |  |
| 4. | **GC Parameters** |  |  |
| **4.1** | **Column** |  |  |
| 4.1.1 | Stainless Steel Packed with 5 % OV-1 on Chromosorb WHP (80-100 mesh) |  |  |
| 4.1.2 | Length: 183 cm |  |  |
| 4.1.3 | I.D.: 3 mm |  |  |
| **4.2** | **Gas** |  |  |
| 4.2.1 | Carrier: Nitrogen 40 mL/min |  |  |
| 4.2.2 | Hydrogen: 45mL/min Air: 450mL/min  |  |  |
| **4.3** | **Temperature**  |  |  |
| 4.3.1. | Oven: 180°C for 3 min. increased by 6°C /min till 240°C and hold for 3 min. |  |  |
| 4.3.2 | Injector: 270°C |  |  |
| 4.3.3. | Detector : 300°C |  |  |
| 4.4. | Injection Volume : 2µL |  |  |
| 4.5. | Range : 1 |  |  |
| 4.6. | Attenuation : -4 |  |  |
| 5. | **Result** |  |  |
| Sample chromatogram no.  |  |
| Standard chromatogram no.  |  |

**6. Calculation:**

|  |  |
| --- | --- |
| Prallethrin content % by mass,  A1 X A4 X M1 = ------------------- X P A2 X A3 X M2  | Where,M1 =Mass in ‘g’ of Prallethrin standard in working standard of Prallethrin and Piperonyl butoxideM2 =Mass in ‘g’ of sample taken for test A1 = Peak area of Prallethrin in the sample solution A2 = Peak area of IS in sample solutionA3= Peak area of Prallethrin in the standard solutionA4= Peak area of IS in the working standard solutionP = Percent purity of Prallethrin in the standard |
| Piperonyl butoxide content % by mass ,  A1 X A4 X M1 = ------------------- X P A2 X A3 X M2   | Where,M1 =Mass in ‘g’ of Piperonyl butoxide standard in working standard of Prallethrin and Piperonyl butoxideM2 = Mass in ‘g’ of sample taken for test A1 = Peak area of Piperonyl butoxide in the sample solution A2 = Peak area of IS in sample solutionA3= Peak area of Piperonyl butoxide in the standard solutionA4= Peak area of IS in the working standard solutionP = Percent purity of Piperonyl butoxide standard |

**Result:**

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Sl. No.** | **Name of test** | **Result** | **Unit** | **Method of Analysis**  |
| 1. | Prallethrin Content |  | % | Customer Method |
| 2. | Piperonyl butoxide Content |  | % | Customer Method |
| Remark / Reference : |

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