Sr. No. in Scope NABL/ NON NABL

**Flow Chart for Analysis of Deltamethrin and Buprofezin in Formulation**

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| **Date of Analysis**  |  |

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| **SI.** **No.** | **Step**  |  **Execution** | **Executed** **By**  |
| 1. | Sample No. |  |  |
| 2. | Name of Sample |
| **3.** | **Procedure** | **R1** | **R2** |  |
| **3.1** | **Preparation of Mobile Phase** |  |  |  |
| 3.1.1 | Mix Isooctane and 1,4-dioxane (0.15% water) in the proportion of 95:5 (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 Make up Phase** |  |  |  |
| 3.2.1 | Mix Isooctane : 1,4-dioxane in the proportion of 80:20 (v/v)  |  |  |  |
| 3.2.2 | Pass through membrane filter under vacuum |  |  |  |
| 3.2.3 | Homogenize the mixture using a magnetic stirrer |  |  |  |
| 3.2.4 | Allow to attain room temperature |  |  |  |
| **3.3** | **Preparation of Internal Standard Solution** |  |  |  |
| 3.3.1 | Weigh 5 g of Di-butyl phthalate (DBP) in 100 ml volumetric flask | g | g |  |
| 3.3.2 | Add 40 ml of make up phase(3.2.4) and mix well | ml | ml |  |
| 3.3.3 | Dilute up to the mark with make up phase (3.2.4) |  |  |  |
| **3.4** | **Preparation of Standard Solution** |  |  |  |
| 3.4.1 | Note the Purity of Deltamethrin standard | % | % |  |
| 3.4.2 | Weigh0.040 g a.i. of the standard Deltamethrin accurately in a 50 ml volumetric flask  | g | g |  |
| 3.4.3 | *Note the serial No. of the balance log book* |  |  |  |
| 3.4.4 | Make up the volume with Make-up phase (3.2.4) (stock A). |  |  |  |
| 3.4.5 | Note the Purity of Buprofezin standard | % | % |  |
| 3.4.6 | Weigh 0.032 g a.i. of the standard Buprofezin accurately in a 50 ml volumetric flask. | g | g |  |
| 3.4.7 | *Note the serial No. of the balance log book* |  |  |  |
| 3.4.8 | Add to it 5 ml of Internal standard solution (3.3.3) |  |  |  |
| 3.4.9 | Add to it 5 ml of Deltamethrin stock A solution (3.4.4) and mix well |  |  |  |
| 3.4.10 | Make up the volume with Make-up phase (3.2.4) |  |  |  |
| **3.5** |  **Preparation of Sample Solution** |  |  |  |
| 3.5.1 | Note the percent active ingredient contents declared on the sample for Deltamethrin and Buprofezin respectively. | % | % |  |
| 3.5.2 | Weigh 0.560 g of sample in 50 ml volumetric flask | g | g |  |
| 3.5.3 | *Note the serial No. of the balance log book* |  |  |  |
| 3.5.4 | Add to it 10 ml of makeup phase (3.2.4) and mix well. |  |  |  |
| 3.5.5 | Add to it 5 ml of Internal Standard solution (3.3.3)and mix well |  |  |  |
| 3.5.6 | Dilute up to the mark with solvent mixture (3.2.4) |  |  |  |
| **4.** | **HPLC Parameters** |  |  |  |
| **4.1** | **Column** |  |  |  |
| 4.1.1 | Stainless Steel Packed with **Silica** |  |  |  |
| 4.1.2 | Length: 250 mm |  |  |  |
| 4.1.3 | I.D.: 4.6 mm |  |  |  |
| 4.1.4 | Particle Size : 5 µ |  |  |  |
| **4.2** | **Mobile Phase** |  |  |  |
| 4.2.1 | Isooctane : 1,4-dioxane (0.15% water) (95 : 5) |  |  |  |
| 4.2.2 | Flow Rate: 0.8 ml/min |  |  |  |
| **4.3** | **Detector:** UV |  |  |  |
| **4.4** | **Wave Length**: 254 nm |  |  |  |
| **4.5** | **Injection Volume:** 20 µl |  |  |  |
| **5.** | **Result** |  |  |  |
| Sample chromatogram no.  |  |  |  |
| Standard chromatogram no.  |  |  |  |

**6. Calculation:**

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| **Deltamethrin** content, % by mass =  A1 x A ‘IS’1 x M1 x 1 -------------------------- XP  A’IS’2 x A2 x M3 x 10 | **Where,**A1 = Peak area of Deltamethrin in the sample solutionA’IS’2 =Peak area of internal standard in the sample solutionA’IS’1 =Peak area of internal standard in the standard solution A2 = Peak area of Deltamethrin in the standard solutionM1  = Mass in ‘g’ of Deltamethrin in the standard solutionM3  = Mass in ‘g’ of sample taken for testP = Percentage purity of Deltamethrin standard10 = Dilution Factor |

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| **Buprofezin** content, % by mass = A3 x A ‘IS’1 x M2 --------------------- XP  A’IS’2 x A4 x M3  | **Where,**A3 = Peak area of Buprofezin in the sample solutionA’IS’2 =Peak area of internal standard in the sample solutionA’IS’1 =Peak area of internal standard in the standard solution A4 = Peak area of Buprofezin in the standard solutionM2  = Mass in ‘g’ of Buprofezin in the standard solutionM3  = Mass in ‘g’ of sample taken for testP = Percentage purity of Buprofezin standard |
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**Result:**

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| **SI. No.** | **Name of test** | **Name of the A.I** | **Result** | **Unit** | **Method of Analysis** |
| 1. | Active ingredient | 1) Deltamethrin |  | % | HPLC Method |
| 2) Buprofezin |  |
| Remark / Reference : |

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