S. No. in Scope NABL / NON NABL

**Flow Chart for Analysis of Phorate Content in Formulation sample**

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

|  |  |  |  |
| --- | --- | --- | --- |
| **Sl. No.** | **Step**  | **Execution** | **Executed By** |
| 1. | Sample No. |  |  |  |
| 2. | Name of Sample |  |  |  |
| 3. | **Procedure** | **R1** | **R2** |  |
| **3.1** | **Preparation of Internal Standard Solution:** |  |  |  |
| 3.1.1 | Weigh 1.0 g of Di-n-butyl Phthalate (DBP) 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 acetone. |  |  |  |
| **3.2** | **Preparation of Standard Solution:**  |  |  |  |
| 3.2.1 | Purity of standard | % | % |  |
| 3.2.2 | Weigh 0.1 g a. i. of standard in a 50 ml volumetric flask. | g | g |  |
| 3.2.3 | Add 5 mL of internal standard solution (3.1.3). | ml | ml |  |
| 3.2.4 | Dilute up to the mark with acetone and shake well. |  |  |  |
| **3.3** | **Preparation of Sample Solution:** |  |  |  |
| 3.3.1 | Note down the percent active ingredient content declared on the sample. |  |  |  |
| 3.3.2 | Weigh 25 g of sample in a 100 ml beaker .  | g | g |  |
| 3.3.3 | *Note down the S. No. of balance log book .* |  |  |  |
| 3.3.4 | Transfer the sample quantitatively with acetone into a 250 mL stoppered conical flask. |  |  |  |
| 3.3.5 | Extract the sample using 150 mL of acetone on a mechanical shaker for 15 min. |  |  |  |
| 3.3.6 | Filter the supernatant through a filter paper into a 250 mL volumetric flask. |  |  |  |
| 3.3.7 | Make the volume up to the mark with acetone and mix well (Stock A). |  |  |  |
| 3.3.8 | Pipette out 10 ml of Stock A (3.3.7) into a 50 ml volumetric flask. |  |  |  |
| 3.3.9 | Add 5 mL of internal standard solution (3.1.3). | ml | ml |  |
| 3.3.10 | Dilute up to the mark with acetone and mix well |  |  |  |
| **4.** | **GC Parameters** |  |  |  |
| 4.1 | **Column** : Packed with 5% SE -30 on Chromosorb WHP (80-100) mesh |  |  |  |
| 4.1.1 | Length: 100 cm  |  |  |  |
| 4.1.2 | I.D: 2 mm |  |  |  |
| **4.2** | **Gas** |  |  |  |
| 4.2.1 | Carrier:Nitrogen: 40 ml/min |  |  |  |
| 4.2.2 |  Hydrogen: 45 ml/min  |  |  |  |
| 4.2.3 | Air : 450 ml/min |  |  |  |
| **4.3** | **Temperature** |  |  |  |
| 4.3.1 | Oven: 1700C  |  |  |  |
| 4.3.2 | Injector: 2100C |  |  |  |
| 4.3.3 | Detector: 2500C |  |  |  |
| **4.4.** | **Injection volume**: 1 µl |  |  |  |
| 5. | **Results** |  |  |  |
| Sample chromatogram no.  |  |  |  |
| Standard chromatogram no. |  |  |  |

**6. Calculation:**

|  |  |
| --- | --- |
| Phorate content, % by mass=  A1 x A’IS’2 x M1 ------------------------ x PA’IS’1 x A2 x M2   | **Where,** A1= Peak area of phorate 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 phorate in the standard solutionM1= Mass in ‘g’ of phorate in standard solutionM2= Mass in ‘g’ of phorate sample taken for testP = Percent purity of phorate standard |

**Result:**

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Sl. No.** | **Name of test** | **Result** | **Unit** | **Method of Analysis**  |
| 1. | Active ingredient |  | % | By GLC |
| Remark / Reference : |

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