Sr. No. in Scope NABL / NON NABL

 **Flow Chart for Analysis of Metribuzin Content 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 3g of Di-butyl Sebacate taken in a 100 mL volumetric Flask. |   |  |
| 3.1.2 | Make up the volume with Acetone. |  |  |
| **3.3 Preparation of Reference Standard** |  |  |
| 3.3.1 | Note the Purity of standard | % |  |
| 3.3.2 | Weigh standard equivalent to 0.25 g of active ingredient 50 ml volumetric flask | g |  |
| 3.3.3 | Add to it 5 ml of Internal Standard solution (3.1.2.)  |  |  |
| 3.3.4 | Dilute up to the mark with Acetone. |  |  |
| 3.3.5 | Shake well to homogenize. |  |  |
|  **3.4 Preparation of Sample Solution** |  |  |
| 3.4.1 | Note the percent active ingredient declared on the sample | % |  |
| 3.4.2 | Weigh accurately 0.357 g sample (equivalent to 0.25g of a.i.) in 50ml volumetric flask. | g |  |
| 3.4.3 | Add to it 5 ml of Internal Standard solution(3.1.2.) |  |  |
| 3.4.4 | Dilute up to the mark with Acetone  |  |  |
| 3.4.5 | Shake well to homogenize. |  |  |
| 4. | **GC Parameters** |  |  |
| **4.1 Column** |  |  |
| 4.1.1 | Stainless Steel Packed with 10% OV-17 on Chromosorb WHP (100-120 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: 210°C |  |  |
| **4.3.2** | Injector: 250°C |  |  |
|  | **4.3.3.** | Detector : 300°C |  |  |
|  | **4.4.** | Injection Volume : 2µL |  |  |
|  | **4.5.** | Range : 1 |  |  |
|  | **4.6.** | Attenuation : -3 |  |  |
| 5. | **Result** |  |  |
| Sample chromatogram no.  |  |
| Standard chromatogram no.  |  |

**6. Calculation:**

|  |  |
| --- | --- |
|  Metribuzin content, A1 X A4 X M1 % by mass =------------------------ X P A2 X A3 X M2   | **Where,**M1 =Mass in ‘g’ of Metribuzin standard M2 =Mass in ‘g’ of sample taken for test  A1 = Peak area of Metribuzin in the sample solution A2 = Peak area of IS in sample solution A3= Peak area of Metribuzin in the standard solution A4= Peak area of IS in the standard solution P = Percent purity of Metribuzin in the standard |

**Result:**

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Sl. No.** | **Name of test** | **Result** | **Unit** | **Method of Analysis**  |
| 1. | Active ingredient |  | % | IS 1332:1992 (Reaffirmed 2007) |
| Remark / Reference : |
| Analyzed by | Name  |  |
| Dated signature |  |
| Checked by | Name  |  |
| Dated signature |  |