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 | |  | | |