Sr. No. in Scope NABL / NON NABL

**Flow chart for estimation of Endosulfan content in formulation sample**

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

|  |  |  |  |
| --- | --- | --- | --- |
| **Sl No.** | **Step**  | **Execution** | **Executed****By** |
|  | Sample No. |  |  |
|  | Name of Sample |  |  |
|  | **Procedure**  |
| 3.1.1 | Weigh accurately equivalent to 0.5 g of sample in 500 ml round Flask  |  g  |  |
| 3.1.2 | Add 100 ml methanol |  |  |
| 3.1.3 | Add 1.5 g (15-16) pellets of NaOH and boiling chips, connect the flask for reflux |  |  |
| 3.1.4 | Boil it for 2 hrs and then cool to room temperature |  |  |
| 3.1.5 | Wash the condenser with 20 ml methanol and 50 ml distilled water. And then remove flask |  |  |
| 3.1.6 | Add 25 ml standard potassium iodate solution (0.025 M) |  |  |
| 3.1.7 | Add 20 ml (10 %) potassium iodide (KI) solution |  |  |
| 3.1.8 | Add 25 ml of (5 N) sulphuric acid and keep the flask in cold water |  |  |
| 3.1.9 | Titrate the liberated iodine against sodium thiosulphate solution to pale yellow colour. Add 1 ml of starch indicator solution (1%), and continue titration |  |  |
| 3.1.10 | End point will be blue to colorless |  |  |
| 3.1.11 | Note the volume of sodium thiosulphate consumed |  ml  |  |
|  | **Preparation of Blank** |   |  |
|  | 4.1.1 | Pipette out 25 ml standard potassium iodate (KIO3) solution in 500 ml conical flask |  |  |
| 4.1.2 | Add 20 ml (10%) potassium iodide (KI) solution |  |  |
| 4.1.3 | Add 25 ml of (5 N) sulphuric acid; the flask should keep in water bath. |  |  |
| 4.1.4 | Titrate the liberated iodine immediately against sodium thiosulphate solution to pale yellow colour. Add 1 ml of starch indicator solution (1%), and continue titration |  |  |
| 4.1.5 | End point will be blue to colorless |  |  |
| 4.1.6 | Note the volume of sodium thiosulphate consumed |  ml  |  |

**5. Calculation:**

 20.35 x t x n

 Endosulfan content, % by mass = -----------------------

 M

**Where,**

t = (V1 – V2) ml

V1 = Volume of standard sodium thiosulphate solution consumed by blank

V2 = Volume of standard sodium thiosulphate solution consumed by sample

M = Mass in ‘g’ of sample taken for test

n = Normality of sodium thiosulphate solution

 20.35 = factor for endosulfan

**Result:**

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Sl .No.** | **Name of test** | **Result** | **Unit** | **Method of Analysis**  |
| 1. | Active ingredient |  | % | Volumetric Analysis (IS- 4344 : 1978) |
| Remark / Reference : |

|  |  |  |
| --- | --- | --- |
| Analysed by | Name  |  |
| Dated signature |  |
| Checked by | Name  |  |
| Dated signature |  |