Sr. No. in Scope NABL / NON NABL

# Flow Chart for Analysis of Malathion Content in Formulation

|  |  |
| --- | --- |
| **Date of Analysis**  |  |
| **S. No.** | **Step** | **Execution** | **Executed By** |
| 1. | Sample No. |  |  |
| 2. | Name of Sample |  |  |
| 2.1 | Sample Description |  |  |
| 3. | **Procedure** |  |  |
| 3.1 | **Preparation of Ferric Reagent solution :** |  |  |
| 3.1.1 |  Weigh 4.0 g of ferric chloride (Fecl3.6H2O) in a 250 mL beaker.  |  |  |
| 3.1.2 | *Note the serial number of balance log book.* |  |  |
| 3.1.3 | Dissolve in 160 mL of concentrated hydrochloric acid.  |  |  |
| 3.1.4 | Transfer the solution to a 500 mL volumetric flask containing around 250 mL of water and make up to the mark with distilled water (Stock F). |  |  |
| 3.1.5 | Pipette out 25 mL of stock F (3.1.4) into a 1000mL volumetric flask and dilute up to the mark with water. |  |  |
| 4 | **Preparation of Standard Solution:** |  |  |
| 4.1 | Note the Purity of Standard. | % |  |
| 4.2 | Weigh 0.1 g a.i of Malathion into a 100 mL volumetric flask.  | g |  |
| 4.3 | *Note the serial number of balance log book.* |  |  |
| 4.4 | Dissolve and make up to the mark with ethyl alcohol. (Stock A) |  |  |
| 4.5 | Pipette out 10 mL of stock A (4.4) into a 100 mL volumetric flask. |  |  |
| 4.6 | Add 1 mL of Acetonitrile and dilute with ethyl alcohol up to the mark (Stock B). |  |  |
| 5. | **Preparation of sample solution:** |  |  |
| 5.1 | Note the percent active ingredient content declared on the sample. | % |  |
| 5.2 | Weigh a quantity of the sample to contain 0.1 g a.i. of Malathion into a 100 mL volumetric flask. | g |  |
| 5.3 | *Note the serial number of balance log book.* |  |  |
| 5.4 | Dissolve and make up to the mark with ethyl alcohol. (Stock C) |  |  |
| 5.5 | Pipette out 10 mL of stock C (5.4) into a 100 mL volumetric flask. |  |  |
| 5.6 | Add 1 mL of Acetonitrile and dilute with ethyl alcohol up to the mark (Stock D). |  |  |
| **6.** | **Procedure:** |  |  |
| 6.1 | Transfer 15 mL each of stock B (4.6) and stock D (5.6) into two different 250mL separating funnels. |  |  |
| 6.2 | Add 2mL of 0.5 N sodium hydroxide solution through pipette and mix well by swirling for 5 to 10 sec |  |  |
| 6.3 | Allow the solution to stand exactly for 2 min. |  |  |
| 6.4 | Immediately add 75mL of ferric reagent and mix well by swirling for 10 sec.  |  |  |
| 6.5 | Allow the solution to stand exactly for 5min. |  |  |
| 6.6 | Exactly after 5 minutes add 50 mL of carbon tetrachloride through volumetric flask into each separating funnel. |  |  |
| 6.7 | Allow the volumetric flask to drain for 1 min. (Standard and sample solutions have to be handled separately from this point onwards). |  |  |
| 6.8 | Add 2mL of 1.5% copper sulphate solution using fast delivery pipette into the separating funnel containing standard solution.  |  |  |
| 6.9 | Immediately after addition of copper sulphate solution start shaking the separating funnel exactly for one minute.  |  |  |
| 6.10 | Measure the absorbance of bottom yellow coloured carbon tetrachloride solution within 2 min. at 420 nm using Carbon tetrachloride solution as a blank. |  |  |
| 6.11 | Repeat the steps from 6.8 to 6.10 for sample solution also.  |  |  |
| 6.12 | Absorbance value for Standard solution is |  |  |
| 6.13 | Absorbance value for sample solution is |  |  |

 **7. Calculation:**

|  |  |
| --- | --- |
|   A x M x P Malathion content, % by mass =……………………… B x m | **Where ,**A = Absorbance of sample solution at 420nmB = Absorbance of standard solution at420nm.M= Mass in grams of pure malathion taken for preparation of standard solution.P = Percentage purity of malathion used for  preparation of standard solutionm= mass in grams of material taken for the preparation of sample solution**.** |

**Result:**

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Sl. No.** | **Name of Test** | **Result** | **Unit** | **Method of Analysis** |
| 1. | Active ingredient (Malathion) |  | % | IS 1832– 1978 |
| Reference/Remarks: |
| Analyzed by | Name  |  |
| Dated signature |  |
| Checked by | Name  |  |
| Dated signature |  |