Sr. No. in Scope NABL / NON NABL

**Flow Chart for Analysis of Methyl Parathion Dusting Powder Formulation**

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

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Sl. No.** | **Step** | **Execution** | | **Executed By** |
| 1. | Sample No. |  |  |  |
| 2.1 | Name of Sample |  |  |  |
| 2.2 | Sample Description |  |  |  |
| 3. | **Procedure** | **R1** | **R2** |  |
| **3.1** | **Preparation of Sample Solution** |  |  |  |
| 3.1.1 | Weight of Sample taken equivalent to 0.8 g A.I in 250 mL Beaker | g | g |  |
| 3.1.2 | Purity of sample | % | % |  |
| 3.1.3 | Add 50 mL of Diethyl Ether and stir-well with a glass rod. |  |  |  |
| 3.1.4 | Leave it to settle (3.1.3). |  |  |  |
| 3.1.5 | Filter (3.1.4) through sintered funnel with suction pump. |  |  |  |
| 3.1.6 | Extract two more times with Ether, each time with 50 mL & all extracts filter through suction. |  |  |  |
| 3.1.7 | Collect the entire ether layer (3.1.6) into 500 mL G.G.flask & evaporate on water bath to 2 mL. |  |  |  |
| 3.1.8 | Evaporate (3.1.7) completely at room temperature. |  |  |  |
| 3.1.9 | Add (3.1.8) 50 mL of Methanol. |  |  |  |
| 3.1.10 | Add (3.1.8) 25 mL of 1 N NaOH (aqueous). |  |  |  |
| 3.1.11 | Keep (3.1.8 to 3.1.10) for refluxion for 1 hour. |  |  |  |
| 3.1.12 | Cool (3.1.11) to room temperature. Rinse the condenser with distilled water and remove the flask. |  |  |  |
| 3.1.13 | Transfer it (3.1.12) into 1000 mL Volumetric Flask quantitatively. |  |  |  |
| 3.1.14 | Make up to the mark with Distilled Water. |  |  |  |
| 3.1.15 | Pipette out 25 mL of above stock solution (3.1.14) into a 250 mL volumetric flask. |  |  |  |
| 3.1.16 | Dilute up to the mark with Distilled Water. |  |  |  |
| 3.1.17 | Pipette out 10 mL of above stock solution (3.1.16) into a 100 mL volumetric flask. |  |  |  |
| 3.1.18 | Dilute up to the mark with Distilled Water. |  |  |  |
| 3.1.19 | Switch on the UV-Visible Spectrophotometer & wait for Stabilization. |  |  |  |
| 3.1.20 | Fill the Cuvettes with Distilled Water as a blank & make the Instrument Absorbance to Zero. |  |  |  |
| 3.1.21 | Fill the Cuvettes with Sample solution (3.1.18) &  measure the absorbance at 400 nm. |  |  |  |
| **3.2** | **Preparation of Standard Solution** |  |  |  |
| 3.2.1 | Weigh 0.8 g of standard Technical Methyl Parathion in 500 mL flat bottom G.G. flask. | g | g |  |
| 3.2.2 | Purity of Standard. | % | % |  |
| 3.2.3 | Add to it (3.2.1) 50 mL of Methanol. |  |  |  |
| 3.2.4 | Add to it (3.2.1) 25 mL of 1 N NaOH (aqueous). |  |  |  |
| 3.2.5 | Keep the mixture (3.2.4) for reflux for 1 hour & cool to room temperature. After cooling wash the condenser with distilled water, collect the washing in the same receiver flask. |  |  |  |
| 3.2.6 | Transfer the contents into 1000 mL volumetric flask & make up to the mark with Distilled Water. |  |  |  |
| 3.2.7 | Pipette out 25 mL of above stock solution (3.2.6) into a 250 mL volumetric flask. |  |  |  |
| 3.2.8 | Dilute up to the mark with Distilled Water. |  |  |  |
| 3.2.9 | Pipette out 10 mL of above stock solution (3.2.8) into a 100 mL volumetric flask. |  |  |  |
| 3.2.10 | Dilute up to the mark with Distilled Water. |  |  |  |
| 3.2.11 | Fill the Cuvettes with Standard solution (3.2.10) & measure the absorbance at 400 nm in UV-Visible Spectrophotometer. |  |  |  |
| 4. | **Preparation of 1% Sodium Carbonate solution** |  |  |  |
| 4.1 | Weigh about 5 g of Na2CO3 & dissolve in 500 mL distilled water. |  |  |  |
| 4.2 | Make the above solution (4.1) into Chilled condition. |  |  |  |
| 5. | **Procedure for Impurities** |  |  |  |
| 5.1 | Weight of Sample taken equivalent to 0.08 g A.I in 250 mL Beaker |  |  |  |
| 5.2 | Add 50 mL of Diethyl Ether and stir-well to dissolve. |  |  |  |
| 5.3 | Leave it to settle (5.2). |  |  |  |
| 5.4 | Filter (5.3) through sintered funnel with suction pump. |  |  |  |
| 5.5 | Extract the Pesticide into ether, for two more times, each time with 50 mL ether & collect all the extracts and filter through suction. |  |  |  |
| 5.6 | Collect the entire ether layer (5.5). |  |  |  |
| 5.7 | Transfer it (5.6) into 250 mL Separating funnel. |  |  |  |
| 5.8 | Extract free p-nitrophenol from (5.7) using 20 mL each time with 1% chilled Sodium Carbonate solution. |  |  |  |
| 5.9 | Collect the Yellow coloured aqueous layer into a 500 mL beaker. |  |  |  |
| 5.10 | Wash the contents in Separating funnel till Sodium Carbonate layer becomes colourless and collect the Yellow coloured layer. |  |  |  |
| 5.11 | Transfer the solution (5.9 & 5.10) into an appropriate volumetric flask and make up to the mark with distilled water. |  |  |  |
| 5.12 | Take the same quantity of Na2CO3 used in extraction of impurities, into an appropriate volumetric flask. |  |  |  |
| 5.13 | Dilute the solution (5.12) up to the mark with distilled water. |  |  |  |
| 5.14 | Use Na2CO3 solution for blank. |  |  |  |
| 5.15 | Fill the Cuvette with sample solution (5.13) and measure the absorbance at 400 nm in UV-Visible Spectrophotometer. |  |  |  |
| 5.16 | Record the absorbance. |  |  |  |

**6. Calculation:**

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| Methyl Parathion content, % by mass = | [ | A2 x M1 | x P] | **—** | [ | A3 x M1 x P | ] |
|  | A1 x M2 | A1 x M3 x F |

**Where,**

M1 =Mass in ‘g’ of Standard.

M2 =Mass in ‘g’ of Sample taken for test.

M3 =Mass in ‘g’ of Sample taken for p-nitrophenol (Impurities) extraction.

A1 = Absorbance of Standard solution.

A2 = Absorbance of Sample solution.

A3 = Absorbance of p-nitrophenol extract.

P = Percentage purity of Standard Methyl Parathion.

F = Dilution factor is 1000; if free p-nitrophenol is diluted to 100 mL.

400; if free p-nitrophenol is diluted to 250 mL.

200; if free p-nitrophenol is diluted to 500 mL.

**Result:**

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Sl. No.** | **Name of test** | **Result** | **Unit** | **Method of Analysis** |
| 1. | Methyl Parathion Content |  | % | IS 2570 : 1980  Reaffirmed 2007 |
| Remark / Reference : | | | | |

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