Sr. No. in Scope NABL / NON NABL **Flow Chart for Analysis of Methyl Parathion content in Technical Concentrate**

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| --- | --- |
| **Date of Analysis**  |  |
| **Sl. No.** | **Step**  | **Execution** | **Executed By**  |
| 1. | Sample No. |  |  |
| 2 | Name of Sample |
| 2.1 | Sample Description |
| **3**. | **Procedure for Estimation of Total Methyl Parathion content:** |
| **3.1.** | **Preparation of Sample Solution:** |  |  |
| 3.1.1. | Note down the percent active ingredient content declared on the sample |  %  |  |
| 3.1.2 | Weigh Sample equivalent to 0.8 g of a.i in a 25 mL Beaker |  g |  |
| 3.1.3 | *Note down the S.no. of balance log book.* |  |  |
| 3.1.4 | Transfer the sample quantitatively to a 500 mL flat bottom GG flask using 50 mL of Methanol. |  |  |
| 3.1.5 | Add 25 mL of 1 N NaOH (aqueous) solution. |  |  |
| 3.1.6 | Keep the solution under reflux for 1 hour. |  |  |
| 3.1.7 | After 1 hr. cool the solution to room temperature. Rinse the condenser with distilled water and remove the flask. |  |  |
| 3.1.8 | Transfer the solution into a 1000 mL Volumetric Flask quantitatively. |  |  |
| 3.1.9 | Make up to the mark with Distilled Water. |  |  |
| 3.1.10 | Pipette out 25 mL of above stock solution (3.1.9) into a 250 mL volumetric flask. |  |  |
| 3.1.11 | Dilute up to the mark with Distilled Water. |  |  |
| 3.1.12 | Pipette out 10 mL of above stock solution (3.1.11) into a 100 mL volumetric flask. |  |  |
| 3.1.13 | Dilute up to the mark with Distilled Water. |  |  |
| 3.1.14 | Switch on the UV-Visible Spectrophotometer & wait for Stabilization. |  |  |
| 3.1.15 | Fill the Cuvettes with Distilled Water as a blank & make the Instrument Absorbance to Zero. |  |  |
| 3.1.16 | Fill the Cuvettes with Sample solution (3.1.13) & measure the absorbance at 400 nm. |  |  |
| 3.1.17 | Absorbance value for total methyl parathion content in the sample is |  |  |
| **3.2** | **Preparation of Standard Solution** |  |  |
| 3.2.1 | Purity of Standard.  | % |  |
| 3.2.2 | Weigh 0.8 g a.i. of standard Technical Methyl Parathion in a 25 mL beaker | g |  |
| 3.2.3 | *Note down the S.no. of balance log book.* |  |  |
| 3.2.4. | Transfer the standard quantitatively to a 500 mL flat bottom GG flask using 50 mL of Methanol. |  |  |
| 3.2.5 | Add to it (3.2.4) 25 mL of 1 N NaOH (aqueous). |  |  |
| 3.2.6 | Keep the solution (3.2.5) 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.  |  |  |
| 3.2.7 | Transfer the contents into 1000 mL volumetric flask & make up to the mark with Distilled Water. |  |  |
| 3.2.8 | Pipette out 25 mL of above stock solution (3.2.7) into a 250 mL volumetric flask. |  |  |
| 3.2.9 | Dilute up to the mark with Distilled Water. |  |  |
| 3.2.10 | Pipette out 10 mL of above stock solution (3.2.9) into a 100 mL volumetric flask. |  |  |
| 3.2.11 | Dilute up to the mark with Distilled Water. |  |  |
| 3.2.12 | Fill the Cuvettes with Standard solution (3.2.11) & measure the absorbance at 400 nm in UV-Visible Spectrophotometer with distilled water as blank. |  |  |
| 3.2.13 | Absorbance value for total methyl parathion (3.2.11) of standard is |  |  |
| **4.** | **Procedure for Impurities** |  |  |
| **4.1** | **Preparation of sample solution:** |  |  |
| 4.1.1 | Weigh Sample equivalent to 0.08 g a.i in 25 mL Beaker | g |  |
| 4.1.2 | *Note down the S.no. of balance log book* |  |  |
| 4.1.3 | Transfer the sample into a 250 mL Separating funnel quantitatively with 50 mL of diethyl ether. |  |  |
| 4.1.4 | Extract free p-nitrophenol using 20 mL each time with 1% chilled Sodium Carbonate solution. |  |  |
| 4.1.5 | Wash the contents of Separating funnel till Sodium Carbonate layer becomes colourless. |  |  |
| 4.1.6 | Collect the Yellow coloured aqueous layer into a 500 mL beaker. |  |  |
| 4.1.7 | Transfer the solution (4.1.6) into an appropriate volumetric flask and make up to the mark with distilled water. |  |  |
| 4.1.8 | Take the same quantity of Na2CO3 used in extraction of impurities, into an appropriate volumetric flask. |  |  |
| 4.1.9 | Dilute the solution up to the mark with distilled water and use this solution as blank solution. |  |  |
| 4.1.10 | Fill the Cuvettes with blank solution (4.1.9) & make the Instrument Absorbance to Zero. |  |  |
| 4.1.10 | Fill the Cuvette with sample solution (4.1.7) and measure the absorbance at 400 nm in UV-Visible Spectrophotometer.  |  |  |
| 4.1.11 | Absorbance value for sample impurities is |  |  |

**5. Calculation:**

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

**Result:**

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