Sr. No. in Scope NABL / NON NABL

**Flow chart for determination of Monocrotophos in formulation sample**

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

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **S. No.** | **Step** | **Execution** | | **Executed By** |
| 1. | Sample No. | **R1** | **R2** |  |
| 2. | Name of Sample |  |  |  |
| **3.** | **Procedure** |  |  |  |
| **3.1** | **Preparation of Sample Stock Solution** |  |  |  |
| 3.1.1 | Note down the percent active ingredient declared on the sample. | % | % |  |
| 3.1.2 | Weigh 3.0 g a. i. of the sample in 100 ml volumetric flask | g | g |  |
| 3.1.3 | *Note down the S. No. of balance log book.* |  |  |  |
| 3.1.4 | Dissolve and make up to the mark with methanol (Stock A) |  |  |  |
| **3.2** | **Hydrolysis of Sample** |  |  |  |
| 3.2.1 | Add through pipette 10 ml of Stock A (3.1.4) into a 250 ml volumetric flask |  |  |  |
| 3.2.2 | Add 10 ml of 5 N NaOH through pipette, shake well and allow to stand for 30 min at 25+5°C. |  |  |  |
| 3.2.3 | Add 1 ml Phenolphthalein indicator solution to 3.2.2 |  |  |  |
| 3.2.4 | Titrate with 1 N HNO3, until pink colour disappears. |  |  |  |
| 3.2.5 | Make up the volume up to the mark with distilled water (Stock B) |  |  |  |
| **3.3** | **Preparation of Standard Stock Solution** |  |  |  |
| 3.3.1 | Percent purity of standard | % | % |  |
| 3.3.2 | Weigh 0.15 g a.i. of standard MMA into a 250 ml volumetric flask | g | g |  |
| 3.3.3 | *Note down the S.No. of balance log book.* |  |  |  |
| 3.3.4 | Make up the volume with methanol and mix well |  |  |  |
| **4.** | **Determination of Total MMA** |  |  |  |
| 4.1 | **Preparation of Blank Solution:** |  |  |  |
| 4.1.1 | Add through pipette 10 ml of sodium nitrate solution into a 100 mL volumetric flask. |  |  |  |
| 4.1.2 | Add through pipette 10 ml of 5% acetic acid solution in methanol in 4.1.1 |  |  |  |
| 4.1.3 | Add 50 ml of methanol in 4.1.1 |  |  |  |
| 4.1.4 | Add through pipette 10 ml of 2.8% anhydrous ferric chloride solution in methanol in 4.1.1 |  |  |  |
| 4.1.5 | Make up the volume with methanol up to the mark and mix well |  |  |  |
| 4.1.6 | Fill the cuvettes with blank solution (4.1.5) after 10 min and make the UV-Vis spectrophotometer instrument reading ‘Zero’ at 544 nm |  |  |  |
| **4.2** | **Preparation of Standard** **Solution :** |  |  |  |
| 4.2.1 | Add through pipette 10 ml of standard MMA solution (3.3.4) in a 100 mL volumetric flask. |  |  |  |
| 4.2.2 | Add through pipette 10 ml of sodium nitrate solution in 4.2.1 |  |  |  |
| 4.2.3 | Add through pipette 10 ml of 5% acetic acid solution in methanol in 4.2.1 |  |  |  |
| 4.2.4 | Add 50 ml of methanol in 4.2.1 |  |  |  |
| 4.2.5 | Add through pipette 10 ml of 2.8% anhydrous ferric chloride solution in methanol in 4.2.1 |  |  |  |
| 4.2.6 | Make up the volume with methanol up to the mark & shake well to homogenize. |  |  |  |
| 4.2.7 | Fill the sample cuvette with (4.2.6) solution & take absorbance at 544 nm in UV-Vis spectrophotometer after 10 min |  |  |  |
| 4.2.8 | Absorbance value for MMA Standard solution (4.2.6) for total MMA is |  |  |  |
| **4.3** | **Preparation of Sample Solution** |  |  |  |
| 4.3.1 | Add through pipette 10 ml sample solution from stock B (3.2.5) into a 100 ml volumetric flask. |  |  |  |
| 4.3.2 | Add through pipette 10 ml of 5% acetic acid solution in methanol in 4.3.1 |  |  |  |
| 4.3.3 | Add 50 ml of methanol in 4.3.1 |  |  |  |
| 4.3.4 | Add through pipette 10 ml of 2.8% anhydrous ferric chloride solution in methanol in 4.3.1 |  |  |  |
| 4.3.5 | Make up the volume with methanol up to the mark & shake well to homogenize. |  |  |  |
| 4.3.6 | Fill the cuvette with (4.3.5) solution & take absorbance at 544 nm in UV-Vis spectrophotometer after 10 min |  |  |  |
| 4.3.7. | Absorbance value for hydrolysed sample solution (4.3.5) for total MMA is |  |  |  |
| **5** | **Determination of Free MMA** |  |  |  |
| **5.1** | **Preparation of Blank Solution:** |  |  |  |
| 5.1.1 | Add through pipette 10 ml of 5% acetic acid solution in methanol in a 100 ml volumetric flask |  |  |  |
| 5.1.2 | Add 50 ml of methanol in 5.1.1 |  |  |  |
| 5.1.3 | Add through pipette 10 ml of 2.8% anhydrous ferric chloride solution in methanol in 5.1.1 |  |  |  |
| 5.1.4 | Make up volume with methanol up to the mark & shake well to homogenize. |  |  |  |
| 5.1.5 | Fill the cuvettes with (5.1.4) solution and make the UV-Vis spectrophotometer instrument reading ‘Zero’ at 544 nm |  |  |  |
| 5.2 | **Preparation of Standard Solution:** |  |  |  |
| 5.2.1 | Add through pipette 10 ml standard MMA (3.3.4) in 100 ml volumetric flask |  |  |  |
| 5.2.2 | Add through pipette 10 ml of 5% acetic acid in methanol in 5.2.1 |  |  |  |
| 5.2.3 | Add 50 ml of methanol in 5.2.1 |  |  |  |
| 5.2.4 | Add through pipette 10 ml of 2.8% anhydrous ferric chloride solution in methanol in 5.2.1 |  |  |  |
| 5.2.5 | Make up volume with methanol up to the mark and mix well |  |  |  |
| 5.2.6 | Fill the sample cuvette with (5.2.5) solution & take absorbance at 544 nm in UV-Vis spectrophotometer immediately |  |  |  |
| 5.2.7 | Absorbance value for MMA Standard solution (5.2.5) for Free MMA is |  |  |  |
| 5.3 | **Preparation of Sample Solution:** |  |  |  |
| 5.3.1 | Add through pipette 10 ml sample solution from stock A (3.1.4) in 100 ml volumetric flask |  |  |  |
| 5.3.2 | Add through pipette 10 ml of 5% acetic acid in methanol in 5.3.1 |  |  |  |
| 5.3.3 | Add 50 ml of methanol in 5.3.1 |  |  |  |
| 5.3.4 | Add through pipette 10 ml of 2.8% anhydrous ferric chloride solution in methanol in 5.3.1 |  |  |  |
| 5.3.5 | Make up the volume with methanol up to the mark and mix well. |  |  |  |
| 5.3.6 | Fill the sample cuvette with (5.3.5) solution & take absorbance at 544 nm in UV-Vis spectrophotometer immediately |  |  |  |
| 5.3.7 | Absorbance value for unhydrolysed sample solution (5.3.5) for Free MMA is |  |  |  |

**6. Calculation:**

|  |  |
| --- | --- |
| **Monocrotophos content, % by mass**  At2 x Ms x P  = 1940 x -------------------- - K  As2 x Mt x 100  **And K is determine by:**  At1 x Ms x P  K= 77.6 X ---------------------  As1 x Mt x 100  Free MMA, % by mass = K / 1.94 | **Where,**  At1= Absorbance of sample in free MMA solution  At2 = Absorbance of sample in total MMA solution  As1 = Absorbance of standard in free MMA  As2 = Absorbance of standard in total MMA  Ms = Mass in ‘g’ of MMA in the standard solution  Mt = Mass in ‘g’ of sample taken for test  P = Percent purity of MMA standard |

**Result:**

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Sl. No.** | **Name of test** | **Result** | **Unit** | **Method of Analysis** |
| 1. | Active ingredient |  | % | IS 8025 : 1990 (Reaffirmmed 2007) |
| Remark / Reference : | | | | |

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