Sr. No. in Scope NABL / NON NABL

**Flow Chart for Analysis of Mancozeb in Formulation**

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

|  |  |  |  |
| --- | --- | --- | --- |
| **Sl. No.** | **Step** | **Execution** | **Executed By** |
| **1.** | Sample No. |  |  |
| **2.** | Name of Sample | | |
| **3.** | **Preparation of 0.1 N Iodine Solution** | | |
| 3.1 | Weigh about 12.69 g of Iodine in a 100 mL beaker. |  |  |
| 3.2 | *Note down the S.No. of the balance log book.* |  |  |
| 3.3 | Transfer the iodine into a 250 mL iodine flask containing 100 mL of 30 % KI solution. |  |  |
| 3.4 | Dissolve the Iodine and transfer quantitatively into a 1000 mL amber coloured volumetric flask and make up to the mark with distilled water. |  |  |
| **4.** | **Standardization of 0.1 N Iodine Solution :** |  |  |
| 4.1 | Pipette out 25 mL of 0.1 N Iodine solution into a 250 ml Iodine flask and dilute it with 75 mL of distilled water. |  |  |
| 4.2 | Titrate the solution against standardized 0.1 N Sodium thiosulphate solution taken in a burette till dark brown colour solution turns into pale yellow colour. |  |  |
| 4.3 | Add 1 mL of starch solution to turn the solution to dark blue. |  |  |
| 4.4 | Continue the titration with 0.1 N Sodium thiosulphate solution till the solution becomes colorless. |  |  |
| **5** | **Procedure** |  |  |
| 5.1 | Take 50 mL of Lead Acetate solution(10% aqueous) into the first absorber bottle. |  |  |
| 5.2 | Take 50 mL and 25 mL of 2N methanolic Potassium hydroxide solution into the second and third absorption bottles respectively. |  |  |
| 5.3 | Take 100-150 mL of 1.1 N Sulphuric acid in the reaction flask. (Sufficient quantity to see that the ‘T’ joint tube dips in H2SO4 solution). |  |  |
| 5.4 | Assemble the apparatus. Connect the opening of the reaction flask to the condenser with an outlet tube connected to three absorbers and which in turn are connected to a water suction pump. Adjust the rate of bubbling in the absorbers to 2-6 bubbles/sec. |  |  |
| 5.5 | Boil the contents of the reaction kettle. |  |  |
| 5.6 | Keep the first absorber with lead acetate solution immersed in a beaker filled with water kept on a hot plate and maintain the temperature of the lead acetate solution at 70°C. |  |  |
| 5.7 | Keep the temperature of methanolic KOH solution in the second and third absorber bottles at 0-10°C throughout the experiment by keeping the absorber bottles in a beaker filled with ice cubes. |  |  |
| 5.8 | Weigh accurately 0.2-0.3 g of a.i. of sample on a filter paper. |  |  |
| 5.9 | *Note down the S.No. of the balance log book.* |  |  |
| 5.10 | Transfer the sample along with filter paper to the reaction flask containing H2SO4 solution under boiling condition at once . |  |  |
| 5.11 | Continue the heating and bubbling for exactly 1 hr and 45 minutes. |  |  |
| 5.12 | After digestion is completed, disconnect the apparatus and quantitatively transfer the methanolic potassium hydroxide solution of the two absorption bottles with distilled water into a 500 mL beaker. (Check for quantintitative transfer by adding phenolphthalein indicator to the absorbtion bottles) |  |  |
| 5.13 | Add 30% acetic acid until the pink color disappears |  |  |
| 5.14 | Add freshly prepared starch solution (1%) and titrate immediately with 0.1 N Iodine solution. |  |  |
| 5.15 | End point of titration is when the colouless solution turns to blue |  |  |
| 5.16 | Titre value for sample is |  |  |

**6. Calculation:**

Normality of Na2S2O3 x Volume of Na2S2O3

1. Normality of Iodine = -----------------------------------------------------------

25

|  |  |
| --- | --- |
| 1. Mancozeb content % by mass =     13.551 X N X V   1. ------------------------  M | **Where,**  V = Volume in ml of standard Iodine solution consumed  N = Noramality of standard Iodine solution  M = Mass in g of the material taken for the test. |

**5. Result:**

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **S.No.** | **Name of test** | **Result** | **Unit** | **Method of Analysis** |
| 1. | Active Ingredient |  | % | IS : 8707-1978  (Reaffirmed 1997) |
| **Remarks/Reference:** | | | | |

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