Sr. No. in Scope NABL / NON NABL

 **Flow chart for analysis of Zinc Phosphide in formulation**

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| --- | --- |
| **Date of Analysis**  |  |
| **Sl. No.** | **Step**  | **Execution** | **Executed By**  |
| 1. | Sample No. |  |  |
| 2. | Name of Sample |
| 3. | **Preparation of Standard 0.5 N Potassium Permanganatesoloution :** |  |  |
| 3.1 | Weigh 15.9 g of Potassium permanganate (KMnO4) in a 250 mL beaker. | g |  |
| 3.2 | *Note down the S.No. of balance log book.* |  |  |
| 3.3 | Dissolve and quantitatively transfer with 1000 mL distilled water to an amber coloured bottle to get 0.5 N solution. |  |  |
| 4. | **Preparation of 0.5 N Oxalic acid Solution :** |  |
| 4.1. | Weigh accurately 15.8 g of AR grade oxalic acid on an analytical balance in a 250 mL beaker and dissolve in about 200ml of water. |  g |  |
| 4.2 | *Note down the S.No. of balance log book.* |  |  |
| 4.3 | Transfer the solution (4.1) to a 500 ml volumetric flask. Add 125ml of sulphuric acid 1:1 (v/v) solution and make up the volume with water . |  |  |
| 5 | **Standardization of 0.5 N KMnO4 Solution:** |  |  |
| 5.1 | Pipette out in 25ml of 0.5 N oxalic acid solution into a 250 mL beaker  | mL |  |
| 5.2 | Warm the solution and maintain the temperature at 60°C by keeping the beaker on a heating mantle . |  |  |
| 5.3 | Add KMnO4 from a burette with continuous stirring till pale pink color appears. |  |  |
| 5.4 | Titre value for Standardization is |  mL |  |
| **6.** | **Procedure:** |  |
| 6.1 | Measure 100 mL of 1 N standard sodium hydroxide solution into the first glass absorption bottle.  |   |  |
| 6.2 | Transfer 100 mL, 50 mL and 50 mL of potassium permanganate solution, with a burette into 2nd , 3rd and 4th absorption bottles respectively. |  |  |
| 6.3 | Take 100 mL of sulphuric acid (10 % aqueous) solution in the reaction flask. (Sufficient quantity to see that the ‘T’ joint tube dips in H2SO4). |  |  |
| 6.4 | Assemble the apparatus pass the N2 gas at least for 30 minutes at 2-3 bubbles/sec so as to displace the air and make the atmosphere inert.(Tighten all the joints between the absorption bottles and make the assembly leak proof). |  |  |
| 6.5 | Note down the percentage active ingredient declared on the sample |  % |  |
| 6.6 | Weigh accurately 0.5 g of sample on an ordinary filter paper. | g |  |
| 6.7 | *Note down the S.No. of balance log book.* |  |  |
| 6.8 | Transfer the sample along with filter paper to the reaction flask at once byopening the lid of the reaction flask slightly and close the lid immediately. |  |  |
| 6.9 | Pass the Nitrogen gas and Keep for reaction for 30 min. at room temperature.  |  |  |
| 6.10 | During the process of setting the assembly, immerse the reaction flask in the water bath.  |  |  |
| 6.11. | After 30 min, increase the temperature of water bath and maintain the temperature of the water bath at 65+ 5 °C. Continue the reaction at least for 1 hr. at this temperature. |  |  |
| 6.12. | Sweep the last traces of phosphine from the flask with more rapid stream of nitrogen for 5 minutes.  |  |  |
| 6.13. | At the end of the reaction disconnect the apparatus and quantitatively transfer the reduced potassium permanganate solution in the three absorption bottles to a 1000 mL beaker. |  |  |
| 6.14. | Rinse the absorption bottles and connecting tubes by adding 200 ml of 0.5 N oxalic acid solution through burette and add the rinsing to the reduced KMnO4 solution. |  |  |
| 6.15. | Warm the contents of the beaker to approximately 60°C and titrate the excess oxalic acid at this temperature with standard 0.5N KMnO4 solution, taken in a burette till pale pink colour end point appears. |  |  |
| 6.16 | Titre value for sample is |  mL |  |

**7. Calculation:**

1. Normality of Oxalic acid = Actual weight of oxalic acid taken X 0.5

 Eq. wt. of Oxalic acid (15.75)

1. Normality of KMnO4 : V1 N1 = V2 N2

Where,

 V1 = Volume of standard potassium permanganate solution consumed.

 V2 = Volume of standard Oxalic acid solution pipette out

 N1 = Noramality of standard potassium permanganate solution

 N2 = Normality of standard Oxalic acid solution

1. Zinc Phosphide content % by mass = 1.613 [(200 +A)N1- 200 N2]
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 M

 Where,

 A = Volume in ml of standard potassium permanganate solution required for the
 titration of excess Oxalic acid

 N1 = Noramality of standard potassium permanganate solution

 N2 = Normality of standard Oxalic acid solution

 M = Mass in g of the material taken for the test.

**8. Result:**

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **S.No.** | **Name of the Test** | **Result** | **Unit** | **Method of Analysis** |
|  | Active ingredient |  | % | IS:1251-1988 (Reaffirmed 2009) |
| Reference/ Remarks: |

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