Sr. No. in Scope NABL / NON NABL

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

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **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 by opening 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 |  |