Sr. No. in Scope NABL / NON NABL

**Flow Chart for analysis of Carbendazim in formulation by Non-Aqueous titration**

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

|  |  |  |  |
| --- | --- | --- | --- |
| **Sl. No.** | **Step** | **Execution** | **Executed By** |
| 1. | Sample No. |  |
| 2. | Name of Sample |  |
| 2.1 | Sample description |  |
| **3.** | **Preparation of Standard Perchloric acid (0 .1 N) :** |  |  |  |
| 3.1 | Take around 500 mL of glacial acetic acid in a 1L volumetric flask. |  |  |  |
| 3.2 | Add 8.5 mL of perchloric acid (70%)  |  |  |  |
| 3.3 | Add 15 mL of acetic anhydride and mix thoroughly. |  |  |  |
| 3.4 | Dilute up to the mark with glacial acetic acid. |  |  |  |
| **4.** | **Standardization of Perchloric acid:** |  |  |  |
| 4.1 | Weigh 0.4-0.5 g of GR grade Potassium Hydrogen Phthalate accurately in a dried 250 mL conical flask | **g** | **g** |  |
| 4.2 | *Note the serial No. of balance logbook* |  |  |  |
| 4.3 | Dissolve in 50 mL of glacial acetic acid and warm the solution to dissolve.  |  |  |  |
| 4.4 | Titrate against 0.1N Perchloric acid solution using 2 drops of crystal violet indicator. |  |  |  |
| 4.5 | End point will be blue to green. |  |  |  |
| 4.6 | Titre value for standardization  | mL | mL |  |
| 4.7. | Titre value for blank is | mL | mL |  |
| 5 | **Procedure:** |  |  |  |
| 5.1 | Note down the percent active ingredient content declared on the sample | % | % |  |
| 5.2 | Weigh sample so as to contain 0.5 g of a.i. in a 250 mL beaker.  | g | g |  |
| 5.3 | *Note the serial No. of balance logbook* |  |  |  |
| 5.4 | Transfer sample to a 60 mL of centrifuge tube. Add 40mL of water and stir well with a rod. |  |  |  |
| 5.5 | Centrifuge @ 2500 rpm for 30 min. and decant the supernatant liquid.  |  |  |  |
| 5.6 | Transfer the sediment quantitatively with 50 mL of glacial acetic acid into dry conical flask |  |  |  |
| 5.7 | Add 10 mL of acetic anhydride. |  |  |  |
| 5.8 | Warm the contents at 50-70°C for 5 minutes and cool it. |  |  |  |
| 5.9 | Filter the contents under suction through sintered glass funnel into a dry flask. |  |  |  |
| 5.10 | Rinse the funnel thrice using 10 mL of glacial acetic acid each time and collect all the rinsings into the conical flask quantitatively. |  |  |  |
| 5.11 | Add 2 drops of crystal violet indicator and titrate with 0.1 N Perchloric acid solution.  |  |  |  |
| 5.12 | End point for titration is from blue to green. |  |  |  |
| 5.13 | Titre value for sample is | mL  | mL  |  |
| **6.** | **Blank titration** |  |  |  |
| 6.1. | Carry out a blank titration without sample, taking 50 mL of glacial acetic acid and 10 mL of acetic anhydride and 2 drops of crystal violet indicator.  |  |  |  |
| 6.2 | End point for titration is from blue to green. |  |  |  |
| 6.3 | Titre value for blank is | mL | mL |  |

**7. Calculation:**

 Wt. of KHP X 1000
 Normality of Perchloric acid = ----------------------------------------------------------------------- =

 (Titre value for sample – Blank) X Eq. wt of KHP(204.22)

|  |  |
| --- | --- |
|   19.12X (V1-V2) × N Active ingredient, = -------------------------- % by mass M  | Where, N=Normality of the standard perchloricacid solution M=Mass in g of the sample taken for test V1=Volume in mL of standard perchloric acid solution consumed for the sample V2 =Volume in mL of standard perchloric acid  solution consumed for blank. |

**Result:**

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Sl. No.** | **Name of Test** | **Result** | **Unit** | **Method of Analysis** |
| 1. | Active ingredient  |  | % | IS: 8446-1991 |
| Remarks: |

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