Sr. No. in Scope NABL / NON NABL

**Flow chart of Suspensibility test for Carbendazim Wettable Powder (WP)
 formulation by Non-aqueous titration**

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| **Date of Analysis**  |  |

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| --- | --- | --- | --- |
| **Sr. No.** | **Step**  | **Execution** | **Executed By**  |
| 1. | Sample No. |  |  |
| 2. | Name of Sample |  |  |
| 3. | **Procedure** |
| **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 thouroughly. |  |  |
| 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 |  |
| 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 |  |
| 4.7. | Titre value for blank is |  mL |  |
| **5.** | **Procedure:** |  |  |
| **5.1** | **Preparation of suspension**  |  |  |
| 5.1.1 | Weigh 5 g sample to prepare suspension in a 100 ml beaker  |  g |  |
| 5.1.2 | *Note the serial No. of balance log book* |  |  |
| 5.1.3 | Add standard hard water 342 ppm (at least twice the mass of the material taken for test) at 30 ± 10C |  |  |
| 5.1.4 | Allow to stand for 15 min. & stir by hand for 30 sec. with a glass rod  |  |  |
| 5.1.5 | Transfer the slurry to the stoppered measuring cylinder (250 ml) and any residue by washing with small quantity of hard water (342 ppm) |  |  |
| 5.1.6 | Add hard water (342 ppm) up to the mark |  |  |
| 5.1.7 | Close the cylinder with the stopper and invert it sharply through 30 complete cycles within 1 min. |  |  |
| 5.1.8 | Allow the cylinder to stand at rest for 30 min. at 30 ± 10C  |  |  |
| 5.1.9 | Withdraw suspension (nine-tenths) from the cylinder within 10 to 15 sec by dipping the nozzle of the glass tube using suction through filtration flask, with out disturbing the bottom 25 mL of suspension. |   |  |
|  5.1.10 | Volume of suspension including sediment at the bottom of the cylinder (one - tenth of the suspension) is |  ml |  |
| **5.2** | **Determination of Active Ingredient** |  |  |
| 5.2.1 | Transfer the bottom 25 mL of suspension into 60 ml centrifuge tube. (Rinse the cylinder with distilled water twice using 20 mL each time). |  |  |
| 5.2.2 | Centrifuge the suspension @ 2500 rpm for 30 min. and decant the supernatant liquid. |  |  |
| 5.2.3 | Transfer the sediment quantitatively with 50 mL of glacial acetic acid into dry conical flask |  |  |
| 5.2.4 | Warm the contents at 50-70°C for 5 minutes and cool it. |  |  |
| 5.2.5 | Filter the contents under suction through sintered glass funnel into a dry flask. |  |  |
| 5.2.6 | Rinse the funnel thrice using 10 mL of glacial acetic acid each time and collect the rinsings quantitatively into a dry conical flsk. |  |  |
| 5.2.7 | Add 10 mL of acetic anhydride and warm the solution. |  |  |
| 5.2.8 | Add 2 drops of crystal violet indicator and titrate with 0.1 N Perchloric acid solution.  |  |  |
| 5.2.9 | End point for titration is from blue to green . |  |  |
|  5.2.10 | Titre value for sample is |  |  |
| 5.3 | **Blank titration** |  |  |
| 5.3.1 | 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.  |  |  |
| 5.3.2 | End point for titration is when the color change from blue to green . |  |  |
| 5.3.3 | Titre value for blank is |  |  |

**4. Calculation:**

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

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

 **ii) Mass in ‘g’ of pesticide present in bottom 25 mL of suspension including sediment is**

 **191.2 X ( titre value for sample – Blank) X N**

 **1000**

N= Normality of perchloric acid solution

1000 (M-m)

**ii )Suspensibility, % by mass** = -----------------------

 9 M

 **Where,**

M = Mass in ‘g’ of pesticide present in the sample taken for the preparation of suspension

m = Mass in ‘g’ of pesticide found in the suspension including the sediment remaining in the

 graduated cylinder

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| --- | --- | --- | --- | --- |
| **Sr. No.** | **Name of test** | **Result** | **Unit** | **Method of Analysis**  |
|  1. | Supensibility |  | % |  **IS : 6940 - 1982** |
| Remark / Reference : |
| Analyzed by | Name  |  |
| Dated signature |  |
| Checked by | Name  |  |
| Dated signature |  |