Sr. No. in Scope NABL / NON NABL

**Flow chart for analysis of Dicofol 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 solutions (0.1 N):** | **R1** | **R2** |  |
| 3.1 | Weigh 16.9 g of AgNO3 and dissolve in water to get 1L solution | g | g |  |
| 3.2 | Weigh 10.5 g of KSCN and dissolve in water to get 1L solution. | g | g |  |
| **4.** | **Standardization of AgNO3:** |  |  |  |
| 4.1 | Weigh 0.1-0.15 g NaCl or KCl GR/AR grade in duplicate into a 250 mL conical flask. | g | g |  |
| 4.2. | Dissolve in 50-70 mL of water |  |  |  |
| 4.3 | Add Potassium chromate indicator (5% aqueous solution). |  |  |  |
| 4.4 | Titrate with AgNO3 solution taken in burette slowly with continuous stirring to pale reddish brown end point. |  |  |  |
| 4.5 | Titre value for the 2 replications | mL | mL |  |
| **5.** | **Standardization of KSCN:** |  |  |  |
| 5.1. | Pipette out 25 mL of standardized 0.1 N AgNO3 solution into a 250 mL conical flask in duplicate. |  |  |  |
| 5.2. | Add 10 mL of 6 N Nitric acid |  |  |  |
| 5.3. | Add 1 mL of Ferric alum indicator |  |  |  |
| 5.4. | Titrate with 0.1 N KSCN taken in burette slowly with continuous stirring to pale reddish brown end point. |  |  |  |
| 5.5. | Titre value for the 2 replications | mL | mL |  |
| 6. | **Procedure** |  |  |  |
| 6.1 | **Preparation of Sample Solution** |  |  |  |
| 6.1.1. | Percent active ingredient declared on the sample | % | % |  |
| 6.1.2 | Weigh Sample equivalent to 0.5 g of active ingredient, transfer to 500ml GG flat bottom flask, quantitatively with 50 mL of ethanol | g | g |  |
| 6.1.3 | Add KOH pellets (1.4 g) to get 50 mL of 0.5 N ethanol |  |  |  |
| 6.1.4 | Place the flask on heating mantle and connect the neck of the flask to water cooled condenser and boil the solution under gentle reflux for 90 min. |  |  |  |
| 6.1.5 | At the end of the time stop heating and allow the flask to cool slightly, rinse the condenser with 25 ml of 95% ethanol. |  |  |  |
| 6.1.6. | Add 2-3 drops phenolphthalein indicator solution, pink colour appears. |  |  |  |
| 6.1.7. | Add sufficient 1:1 Nitric acid solution to turn the solution colourless. |  |  |  |
| 6.1.8. | Add an additional 5 ml of 1:1 Nitric acid and 50ml of the standard silver nitrate solution through pipette or burette. Thoroughly mix the contents of the conical flask. |  |  |  |
| 6.1.9. | Add 5ml of nitrobenzene to the flask, stir vigorously and add 1 mL of ferric alum indicator. |  |  |  |
| 6.1.10 | Titrate the excess of silver nitrate with the standard potassium thiocyanate solution with continuous stirring, until the appearance of faint but permanent brick red end point. |  |  |  |
| 6.1.11 | Titre value for sample is | mL | mL |  |
| **6.2.** | **Reagent Blank** |  |  |  |
| 6.2.1. | Follow the steps from 6.1.2 to 6.1.10, taking all the reagents except sample for reagent blank. |  |  |  |
| 6.2.2 | Titre value for reagent blank is | mL | mL |  |

**7. Calculation:**

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| Wt. of NaCl X 1000  Normality of AgNO3 = ------------------------------------------------------   Volume of AgNO3 consumed X 58.46 |
| Normality of AgNO3 X volume of AgNO3 taken  Normality of KSCN = ------------------------------------------------------------------------  volume of KSCN consumed |

|  |
| --- |
| (mL of KSCN consumed for Blank)-(mL of KSCN consumed for sample) XN X12.351  Active ingredient content, = ----------------------------------------------------------------------------------------------------------  % by mass Weight of sample (g) |
| Where,  N= Normality of KSCN solution |

**8. Result:**

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Sl. No.** | **Name of test** | **Result** | **Unit** | **Method of Analysis** |
| 1. | Active ingredient |  | % (w/w) | I IS: 5278-1969 |
| Remark / Reference : | | | | |

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