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PESTICIDE FORMULATION & RESIDUE ANALYTICAL CENTRE, PMD, NIPHM, HYDERABAD

Sr. No. in Scope

NABL / NON NABL

# Flow Chart for analysis of Alpha Naphthyl Acetic Acid in formulation

|         |   | Date o     | Date of Analysis |                |  |  |
|---------|---|------------|------------------|----------------|--|--|
| SI. No. | Step  | Execu      | tion             | Executed<br>By |  |  |
| 1.      | Sample No.  |            |                  |                |  |  |
| 2.      | Name of Sample  |            |                  |                |  |  |
| 2.1     | Sample description  |            |                  |                |  |  |
| 3.      | Procedure   | <b>R</b> 1 | R <sub>2</sub>   |                |  |  |
| 3.1     | Preparation of Standard solutions 0.1 N NaOH  |            |                  |                |  |  |
| 3.1.1   | Weigh 4 g of NaOH and dissolve in water to get 1L standard solution.                                  | g          | g                |                |  |  |
| 3.1.2   | Note the serial No. in balance logbook  |            |                  |                |  |  |
| 4.      | Standardization of NaOH:  |            |                  |                |  |  |
| 4.1     | Weigh 0.42-0.45 g of GR grade Potassium Hydrogen<br>Phthalate in a 250 mL conical flask               | g          | g                |                |  |  |
| 4.2     | Note the serial No. of balance logbook  |            |                  |                |  |  |
| 4.3     | Dissolve in 40-50 mL of H <sub>2</sub> 0  |            |                  |                |  |  |
| 4.4     | Titrate against 0.1N NaOH solution in burette using phenolphthalein indicator                         |            |                  |                |  |  |
| 4.5     | End point will be colorless to pale pink  |            |                  |                |  |  |
| 4.6     | Titre value for standardization   | mL         | mL               |                |  |  |
| 5.      | Preparation of Buffer solution  |            |                  |                |  |  |
| 5.1     | Weigh 2.035 g of Citric acid and 2.924 g of Disodium hydrogen phosphate.                              | g          | g                |                |  |  |
| 5.2     | Note the serial No. of balance logbook  |            |                  |                |  |  |
| 5.3     | Dissolve in water and make up the volume to 200 mL with water   |            |                  |                |  |  |
| 6.      | Procedure for Alpha NAA   |            |                  |                |  |  |
| 6.1     | Weigh about 10 g of sample.   | g          | g                |                |  |  |
| 6.2     | Note the serial No. of balance logbook  |            |                  |                |  |  |
| 6.3     | Evaporate the solvent on a water bath to get dry residue of the sample.                               |            |                  |                |  |  |
| 6.4     | Add 5 mL water to dissolve the dry residue.   |            |                  |                |  |  |
| 6.5     | Add 50 mL of buffer solution (5.3) and stir.  |            |                  |                |  |  |
| 6.6     | Transfer the solution to a separating funnel quantitatively using 50 mL of Diethyl ether              |            |                  |                |  |  |
| 6.7     | Swirl the mixture and allow the layers to separate.   |            |                  |                |  |  |
| 6.8     | Transfer the aqueous layer to another 250 mL separating funnel and extract with 25 mL of ether twice. |            |                  |                |  |  |

| Name of the Laboratory : Pesticide Formulation & Residue Analytical Centre, PMD, NIPHM, Hyderabad |      |            |                                    |     |  |  |  |
|---|------|------------|------------------------------------|-----|--|--|--|
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| Revision No.  | :    | 02         | Issue Date                         | ••• | 15/07/2013   |  |  |
| Revision Date   | :    | 26/03/2014 | Next Revision Date                 | ••• | 26/03/2016   |  |  |
| Prepared By   |      |            | Checked By                         |     | Approved & Issued By                                   |  |  |
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| 6.9  | Collect the ether layers of all the three separating funnels into |    |    |  |
|------|---|----|----|--|
|      | one.  |    |    |  |
| 6.10 | Wash the ether layer with water to remove any traces of mineral   |    |    |  |
|      | acid  |    |    |  |
| 6.11 | Transfer ether extract to a 500 mL beaker and evaporate to        |    |    |  |
|      | dryness on water bath.  |    |    |  |
| 6.12 | Dissolve the residue in 50 mL neutralized methanol and            |    |    |  |
|      | titrate with standard0.1 N NaOH solution using                    |    |    |  |
|      | phenolphthalein indicator   |    |    |  |
| 6.13 | End point for titration is when the color changes from            | mL | mL |  |
|      | colorless to pale pink.   |    |    |  |

#### 7. Calculation:

Wt. of KHP X 1000

Normality of NaOH =

Volume of NaOH consumed X Eq. wt of KHP(204.22)

| Active ingredient, = | V × N ×18.62<br>M | Where,<br>V=Volume in mL of standard NaOH solution consumed<br>for the sample<br>N=Normality of the standard NaOH solution<br>M=Mass in g of the sample taken for test |
|----------------------|-------------------|--|
|                      |                   |  |

| ]   | Result:                 |                 |           |                                 |                                     |              |  |                    |  |
|---|-------------------------|-----------------|-----------|---------------------------------|-------------------------------------|--------------|--|--------------------|--|
| Sl. No.   | N                       | Name of Test    |           |                                 | Result                              |              | Unit   | Method of Analysis |  |
| 1.  | Active ingredient (Alph |                 |           | na NAA)                         |                                     |              | %  | IS 13138:1991      |  |
| Reference   | in Daily wor            | kbook           |           |                                 |                                     |              |  |                    |  |
| Analyzed by                                       |                         | Nam             | e         |                                 |                                     |              |  |                    |  |
|   |                         | Dated signature |           | ture                            |                                     |              |  |                    |  |
| Checked by  |                         | Name            |           |                                 |                                     |              |  |                    |  |
|   |                         | Dated signature |           | ture                            |                                     |              |  |                    |  |
| Name of the L                                     | aboratory :             | Pesti           | cide Forr | nulation & R                    | esidue Analytical Centre, P         | MD, NI       | PHM, Hyde  | rabad              |  |
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