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

Sr. No. in Scope NABL / NON NABL

Flow Chart for Analysis of Methyl Parathion content in Technical Concentrate

		Date of Analysis	
Sl. No.	**Step**	**Execution**	**Executed By**
1.	Sample No.		
2	Name of Sample		
2.1	Sample Description		
3.	**Procedure for Estimation of Total Methyl Parathion content:**		
3.1.	**Preparation of Sample Solution:**		
3.1.1.	Note down the percent active ingredient content declared on the sample	%	
3.1.2	Weigh Sample equivalent to 0.8 g of a.i in a 25 mL Beaker	g	
3.1.3	*Note down the S.no. of balance log book.*		
3.1.4	Transfer the sample quantitatively to a 500 mL flat bottom GG flask using 50 mL of Methanol.		
3.1.5	Add 25 mL of 1 N NaOH (aqueous) solution.		
3.1.6	Keep the solution under reflux for 1 hour.		
3.1.7	After 1 hr. cool the solution to room temperature. Rinse the condenser with distilled water and remove the flask.		
3.1.8	Transfer the solution into a 1000 mL Volumetric Flask quantitatively.		
3.1.9	Make up to the mark with Distilled Water.		
3.1.10	Pipette out 25 mL of above stock solution (3.1.9) into a 250 mL volumetric flask.		
3.1.11	Dilute up to the mark with Distilled Water.		
3.1.12	Pipette out 10 mL of above stock solution (3.1.11) into a 100 mL volumetric flask.		
3.1.13	Dilute up to the mark with Distilled Water.		
3.1.14	Switch on the UV-Visible Spectrophotometer & wait for Stabilization.		
3.1.15	Fill the Cuvettes with Distilled Water as a blank & make the Instrument Absorbance to Zero.		
3.1.16	Fill the Cuvettes with Sample solution (3.1.13) & measure the absorbance at 400 nm.		
3.1.17	Absorbance value for total methyl parathion content in the sample is		
3.2	**Preparation of Standard Solution**		
3.2.1	Purity of Standard.	%	
3.2.2	Weigh 0.8 g a.i. of standard Technical Methyl Parathion in a 25 mL beaker	g	
3.2.3	*Note down the S.no. of balance log book.*		
3.2.4.	Transfer the standard quantitatively to a 500 mL flat bottom GG flask using 50 mL of Methanol.		

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3.2.5	Add to it (3.2.4) 25 mL of 1 N NaOH (aqueous).		
3.2.6	Keep the solution (3.2.5) for reflux for 1 hour & cool to room temperature. After cooling wash the condenser with distilled water, collect the washing in the same receiver flask.		
3.2.7	Transfer the contents into 1000 mL volumetric flask & make up to the mark with Distilled Water.		
3.2.8	Pipette out 25 mL of above stock solution (3.2.7) into a 250 mL volumetric flask.		
3.2.9	Dilute up to the mark with Distilled Water.		
3.2.10	Pipette out 10 mL of above stock solution (3.2.9) into a 100 mL volumetric flask.		
3.2.11	Dilute up to the mark with Distilled Water.		
3.2.12	Fill the Cuvettes with Standard solution (3.2.11) & measure the absorbance at 400 nm in UV-Visible Spectrophotometer with distilled water as blank.		
3.2.13	Absorbance value for total methyl parathion (3.2.11) of standard is		
4.	**Procedure for Impurities**		
4.1	**Preparation of sample solution:**		
4.1.1	Weigh Sample equivalent to 0.08 g a.i in 25 mL Beaker	g	
4.1.2	*Note down the S.no. of balance log book*		
4.1.3	Transfer the sample into a 250 mL Separating funnel quantitatively with 50 mL of diethyl ether.		
4.1.4	Extract free p-nitrophenol using 20 mL each time with 1% chilled Sodium Carbonate solution.		
4.1.5	Wash the contents of Separating funnel till Sodium Carbonate layer becomes colourless.		
4.1.6	Collect the Yellow coloured aqueous layer into a 500 mL beaker.		
4.1.7	Transfer the solution (4.1.6) into an appropriate volumetric flask and make up to the mark with distilled water.		
4.1.8	Take the same quantity of Na_2CO_3 used in extraction of impurities, into an appropriate volumetric flask.		
4.1.9	Dilute the solution up to the mark with distilled water and use this solution as blank solution.		
4.1.10	Fill the Cuvettes with blank solution (4.1.9) & make the Instrument Absorbance to Zero.		
4.1.10	Fill the Cuvette with sample solution (4.1.7) and measure the absorbance at 400 nm in UV-Visible Spectrophotometer.		
4.1.11	Absorbance value for sample impurities is		

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5. Calculation:

Methyl Parathion content,
% by mass =

$$\left[\frac{A_2 \times M_1}{A_1 \times M_2} \times P\right] - \left[\frac{A_3 \times M_1 \times P}{A_1 \times M_3 \times F}\right]$$

Where,

M_1 =Mass in 'g' of Standard.
M_2 = Mass in 'g' of Sample taken for test.
M_3 = Mass in 'g' of Sample taken for p-nitrophenol (Impurities) extraction.
A_1 = Absorbance of Standard solution.
A_2 = Absorbance of Sample solution.
A_3 = Absorbance of p-nitrophenol extract.
P = Percentage purity of Standard Methyl Parathion.
F = Dilution factor

Result:

Sl. No.	Name of test	Result	Unit	Method of Analysis
1.	Methyl Parathion Content		%	IS 2570 : 1980 Reaffirmed 2007
Remark / Reference :				
Analyzed by	Name			
	Dated signature			
Checked by	Name			
	Dated signature			

Name of the Laboratory : **Pesticide Formulation & Residue Analytical Centre, PMD, NIPHM, Hyderabad**						
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