

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

Sr. No. in Scope

NABL / NON NABL

Flow chart for determination of Monocrotophos in formulation sample

Date of Analysis

S. No.	Step	Execution		Executed By
		R ₁	R ₂	
1.	Sample No.			
2.	Name of Sample			
3.	Procedure			
3.1	Preparation of Sample Stock Solution			
3.1.1	Note down the percent active ingredient declared on the sample.	%	%	
3.1.2	Weigh 3.0 g a. i. of the sample in 100 ml volumetric flask	g	g	
3.1.3	<i>Note down the S. No. of balance log book.</i>			
3.1.4	Dissolve and make up to the mark with methanol (Stock A)			
3.2	Hydrolysis of Sample			
3.2.1	Add through pipette 10 ml of Stock A (3.1.4) into a 250 ml volumetric flask			
3.2.2	Add 10 ml of 5 N NaOH through pipette, shake well and allow to stand for 30 min at 25±5°C.			
3.2.3	Add 1 ml Phenolphthalein indicator solution to 3.2.2			
3.2.4	Titrate with 1 N HNO ₃ , until pink colour disappears.			
3.2.5	Make up the volume up to the mark with distilled water (Stock B)			
3.3	Preparation of Standard Stock Solution			
3.3.1	Percent purity of standard	%	%	
3.3.2	Weigh 0.15 g a.i. of standard MMA into a 250 ml volumetric flask	g	g	
3.3.3	<i>Note down the S.No. of balance log book.</i>			
3.3.4	Make up the volume with methanol and mix well			
4.	Determination of Total MMA			
4.1	Preparation of Blank Solution:			
4.1.1	Add through pipette 10 ml of sodium nitrate solution into a 100 mL volumetric flask.			
4.1.2	Add through pipette 10 ml of 5% acetic acid solution in methanol in 4.1.1			
4.1.3	Add 50 ml of methanol in 4.1.1			
4.1.4	Add through pipette 10 ml of 2.8% anhydrous ferric chloride solution in methanol in 4.1.1			
4.1.5	Make up the volume with methanol up to the mark and mix well			
4.1.6	Fill the cuvettes with blank solution (4.1.5) after 10 min and make the UV-Vis spectrophotometer instrument reading 'Zero' at 544 nm			

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

Document No. : FC-PF-202 Document Name : Flow chart for determination of Monocrotophos content, % by mass

Revision No. : 02 Issue Date : 01/07/2011

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Prepared By

Checked By

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(Director PM & Quality Manager)

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4.2	Preparation of Standard Solution :			
4.2.1	Add through pipette 10 ml of standard MMA solution (3.3.4) in a 100 mL volumetric flask.			
4.2.2	Add through pipette 10 ml of sodium nitrate solution in 4.2.1			
4.2.3	Add through pipette 10 ml of 5% acetic acid solution in methanol in 4.2.1			
4.2.4	Add 50 ml of methanol in 4.2.1			
4.2.5	Add through pipette 10 ml of 2.8% anhydrous ferric chloride solution in methanol in 4.2.1			
4.2.6	Make up the volume with methanol up to the mark & shake well to homogenize.			
4.2.7	Fill the sample cuvette with (4.2.6) solution & take absorbance at 544 nm in UV-Vis spectrophotometer after 10 min			
4.2.8	Absorbance value for MMA Standard solution (4.2.6) for total MMA is			
4.3	Preparation of Sample Solution			
4.3.1	Add through pipette 10 ml sample solution from stock B (3.2.5) into a 100 ml volumetric flask.			
4.3.2	Add through pipette 10 ml of 5% acetic acid solution in methanol in 4.3.1			
4.3.3	Add 50 ml of methanol in 4.3.1			
4.3.4	Add through pipette 10 ml of 2.8% anhydrous ferric chloride solution in methanol in 4.3.1			
4.3.5	Make up the volume with methanol up to the mark & shake well to homogenize.			
4.3.6	Fill the cuvette with (4.3.5) solution & take absorbance at 544 nm in UV-Vis spectrophotometer after 10 min			
4.3.7.	Absorbance value for hydrolysed sample solution (4.3.5) for total MMA is			
5	Determination of Free MMA			
5.1	Preparation of Blank Solution:			
5.1.1	Add through pipette 10 ml of 5% acetic acid solution in methanol in a 100 ml volumetric flask			
5.1.2	Add 50 ml of methanol in 5.1.1			
5.1.3	Add through pipette 10 ml of 2.8% anhydrous ferric chloride solution in methanol in 5.1.1			
5.1.4	Make up volume with methanol up to the mark & shake well to homogenize.			
5.1.5	Fill the cuvettes with (5.1.4) solution and make the UV-Vis spectrophotometer instrument reading 'Zero' at 544 nm			

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5.2	Preparation of Standard Solution:			
5.2.1	Add through pipette 10 ml standard MMA (3.3.4) in 100 ml volumetric flask			
5.2.2	Add through pipette 10 ml of 5% acetic acid in methanol in 5.2.1			
5.2.3	Add 50 ml of methanol in 5.2.1			
5.2.4	Add through pipette 10 ml of 2.8% anhydrous ferric chloride solution in methanol in 5.2.1			
5.2.5	Make up volume with methanol up to the mark and mix well			
5.2.6	Fill the sample cuvette with (5.2.5) solution & take absorbance at 544 nm in UV-Vis spectrophotometer immediately			
5.2.7	Absorbance value for MMA Standard solution (5.2.5) for Free MMA is			
5.3	Preparation of Sample Solution:			
5.3.1	Add through pipette 10 ml sample solution from stock A (3.1.4) in 100 ml volumetric flask			
5.3.2	Add through pipette 10 ml of 5% acetic acid in methanol in 5.3.1			
5.3.3	Add 50 ml of methanol in 5.3.1			
5.3.4	Add through pipette 10 ml of 2.8% anhydrous ferric chloride solution in methanol in 5.3.1			
5.3.5	Make up the volume with methanol up to the mark and mix well.			
5.3.6	Fill the sample cuvette with (5.3.5) solution & take absorbance at 544 nm in UV-Vis spectrophotometer immediately			
5.3.7	Absorbance value for unhydrolysed sample solution (5.3.5) for Free MMA is			

6. Calculation:**Monocrotophos content, % by mass**

$$= 1940 \times \frac{At_2 \times Ms \times P}{As_2 \times Mt \times 100} - K$$

Where,

At₁ = Absorbance of sample in free MMA solution
 At₂ = Absorbance of sample in total MMA solution
 As₁ = Absorbance of standard in free MMA
 As₂ = Absorbance of standard in total MMA
 Ms = Mass in 'g' of MMA in the standard solution
 Mt = Mass in 'g' of sample taken for test
 P = Percent purity of MMA standard

And K is determine by:

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$$K = 77.6 \times \frac{A_{t_1} \times M_s \times P}{A_{s_1} \times M_t \times 100}$$

$$\text{Free MMA, \% by mass} = K / 1.94$$

Result:

Sl. No.	Name of test	Result	Unit	Method of Analysis
1.	Active ingredient		%	IS 8025 : 1990 (Reaffirmed 2007)

Remark / Reference :

Analyzed by	Name	
	Dated signature	
Checked by	Name	
	Dated signature	

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