

## PESTICIDE FORMULATION &amp; RESIDUE ANALYTICAL CENTRE, PMD, NIPHM, HYDERABAD

Sr. No. in Scope

NABL /NON NABL

**Flow chart for analysis of Allethrin content in formulation sample**

		Date of Analysis	
Sl. No.	Step	Execution	Executed By
1.	Sample No.		
2.	Name of Sample		
3	<b>Procedure</b>		
	<b>3.1 Preparation of Internal Standard</b>		
3.1.1	Weight of the Di-butyl Pthalate taken into a 50 ml volumetric flask	g	
3.1.2	Dissolve and dilute up to the mark with toluene		
	<b>3.2 Preparation of Standard</b>		
3.2.1	Weight of the standard taken into 25 ml volumetric flask (stock solution)	g	
3.2.2	Purity of standard	%	
3.2.3	Volume of stock solution (3.2.1) taken into another 25 ml volumetric flask	ml	
3.2.4	Add internal standard solution (3.1.2)	ml	
3.2.5	Dilute up to the mark with toluene		
	<b>3.3 Preparation of Sample</b>		
3.3.1	Weight of the sample taken into 250 ml stopper conical flask	g	
3.3.2	Volume of toluene added to the sample	ml	
3.3.3	Volume of formic acid added to the sample	ml	
3.3.4	Shaking time (first)	min	
3.3.5	Amount of anhydrous sodium sulphate added	g	
3.3.6	Amount of activated charcoal added		
3.3.7	Shaking time (second)	min	
3.3.8	Extract of the sample solution is collected through filtration using buchner funnel		
3.3.9	Internal standard solution (3.1.2) taken into a 25 ml volumetric flask	ml	
3.3.10	Dilute up to the mark with filtrate sample solution		
4.	<b>GC Parameters</b>		
	<b>4.1 Column</b>		
4.1.1	Stainless steel column, packed with 5% OV-1 on chromosorb WHP (80 - 100) mesh		
4.1.2	Length: 180 cm		
4.1.3	I.D.: 1/8" or 2 mm		
	<b>4.2 Gas</b>		
4.2.1	Carrier: Nitrogen: 35 ml/min		
4.2.2	Hydrogen: 45 ml/min		

Name of the Laboratory : <b>Pesticide Formulation &amp; Residue Analytical Centre, PMD, NIPHM, Hyderabad</b>			
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Revision No.	:	00	Issue Date : 1.04.2012
Revision Date	:	1.04.2014	Next Revision Date : 1.04.2016
Prepared By		Checked By	Approved & Issued By
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	4.2.3	Air: 450 ml/min		
	<b>4.3 Temperature</b>			
	4.3.1	Oven: 195 <sup>o</sup> C		
	4.3.2	Injector: 230 <sup>o</sup> C		
	4.3.3	Detector: 260 <sup>o</sup> C		
	<b>4.4 Injection volume:</b>		2 µl	
	<b>4.5 Range:</b>		1	
	<b>4.6 Attenuation:</b>		-5	
5.	<b>Results</b>			
	Sample chromatogram no.			
	Standard chromatogram no.			

**6. Calculation:**

$$\text{Allethrin content, \% by mass} = \frac{A_1 \times A'IS'_2 \times M_1}{A'IS'_1 \times A_2 \times M_2} \times P$$

**Where,**

- $A_1$  = Peak area of allethrin in the sample solution  
 $A'IS'_1$  = Peak area of internal standard in the sample solution  
 $A'IS'_2$  = Peak area of internal standard in the standard solution  
 $A_2$  = Peak area of allethrin in the standard solution  
 $M_1$  = Mass in 'g' of standard allethrin in the standard solution  
 $M_2$  = Mass in 'g' of allethrin sample taken for test  
 $P$  = Percent purity of allethrin standard

**Result:**

Sl. No.	Name of test	Result	Unit	Method of Analysis
1.	Active ingredient		%	

Remark / Reference :

Analyzed by	Name	
	Dated signature	
Checked by	Name	
	Dated signature	

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