

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

Sr. No. in Scope

NABL /NON NABL

Flow chart for analysis of D-Trans Allethrin and Piperonyl butoxide content in aerosol sample

		Date of Analysis	
Sl. No.	Step	Execution	Executed By
1.	Sample No.		
2.	Name of Sample		
3.	Procedure		
	3.1 Estimation of premix & aerosol quantity		
3.1.1	Weight of the container along with aerosol (W_1)	g	
3.1.2	Keep the container in deep freezer overnight		
3.1.3	Puncture the container near neck using sharp nail & allow propellant (LPG) to escape for 2 hours. Sonicate for 10 min & record the weight (W_2)	g	
3.1.4	De-cramp the container and transfer the premix in a clean and dry stopper reagent bottle		
3.1.5	Wash the container with acetone thoroughly and dry it. Record the weight of the empty container (W_3)	g	
3.1.6	Weight of LPG (W_4) = ($W_1 - W_2$)	g	
3.1.7	Weight of Aerosol (W_5) = ($W_1 - W_3$)	g	
3.1.8	Weight of premix (W_6) = ($W_5 - W_4$)	g	
	3.2 Preparation of internal standard solution		
3.2.1	Weight of the Di-butyl phthalate taken into a 25 ml volumetric flask	g	
3.2.2	Dissolve in acetone and dilute up to the mark with the same solvent		
	3.3 Preparation of D- Trans allethrin standard solution		
3.3.1	Weight of the D-Trans allethrin taken into 25 ml volumetric flask	g	
3.3.2	Purity of D- Trans allethrin standard	%	
3.3.3	Dissolve in acetone and dilute up to the mark with the same solvent		
	3.4 Preparation of working standard solution		
3.4.1	Weight of piperonyl butoxide (PBO) taken in 10 ml volumetric flask	g	
3.4.2	Purity of piperonyl butoxide (PBO)	%	
3.4.3	Add D-Trans allethrin standard solution (3.3.2)	ml	
3.4.4	Add internal standard solution (3.2.2)	ml	

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Document No.	:	FC-PF-232	Document Name	:	Flow chart for analysis of D-trans allethrin and piperonyl butoxide content, % by mass
Revision No.	:	00	Issue Date	:	05.10.2012
Revision Date	:	05.10.2014	Next Revision Date	:	05.10.2016
Prepared By		Checked By		Approved & Issued By	
Ms. M. Jaya Devi (Deputy Technical Manager)		Mr. C.V. Rao (Technical Manager)		Dr. Abhay Ekbote (Director PM & Quality Manager)	

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	3.4.5	Dissolve in acetone and dilute up to the mark with the same solvent		
	3.5 Preparation of Sample solution			
	3.5.1	Weight of the sample taken into 25 ml volumetric flask	g	
	3.5.2	Dissolve in acetone and dilute up to the mark with the same solvent		
4.	GC Parameters			
	4.1 Column			
	4.1.1	Stainless steel column, packed with 5% OV-1 on chromosorb WHP (80 - 100) mesh		
	4.1.2	Length: 1.8 m		
	4.1.3	I.D.: 1/8"		
	4.2 Gas			
	4.2.1	Carrier: Nitrogen: 30 ml/min		
	4.2.2	Hydrogen: 45 ml/min		
	4.2.3	Air: 450 ml/min		
	4.3 Temperature			
	4.3.1	Oven: 180°C for 5 min		
		@ 15°C/min to 230°C hold for 7 min		
		@ 20°C/min to 250°C hold for 3 min		
	4.3.2	Injector: 250°C		
	4.3.3	Detector: 260°C		
	4.4 Injection volume:		2 µl	
	4.5 Range:		1	
	4.6 Attenuation:		-2	
5.	Results			
	Sample chromatogram no.			
	Standard chromatogram no.			

6. Calculation:

i) For D-Trans Allethrin

$$\text{D-Trans allethrin content in premix, \% by mass} = \frac{A_2 \times A_3 \times M_1}{A_4 \times A_1 \times M_2} \times P \times df$$

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Sample no

Where,

- A_2 = Peak area of D-trans allethrin in the sample solution
 A_4 = Peak area of internal standard in the sample solution
 A_3 = Peak area of internal standard in the standard solution
 A_1 = Peak area of D-trans allethrin in the standard solution
 M_1 = Mass in 'g' of D-trans allethrin standard
 M_2 = Mass in 'g' of sample (premix) taken for test
df = dilution factor (1/5)
P = Percent purity of D-trans allethrin standard

$$\text{D-trans allethrin content in aerosol, \% by mass} = \frac{\% \text{ A.I in premix} \times \text{Weight of premix (W}_6\text{)}}{\text{Weight of Aerosol (W}_5\text{)}}$$

ii) For Piperonyl butoxide (PBO)

$$\text{Piperonyl butoxide (PBO) content in premix, \% by mass} = \frac{A_2 \times A_3 \times M_1}{A_4 \times A_1 \times M_2} \times P$$

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- A_2 = Peak area of pipernoyl butoxide (PBO) in the sample solution
 A_4 = Peak area of internal standard in the sample solution
 A_3 = Peak area of internal standard in the standard solution
 A_1 = Peak area of pipernoyl butoxide (PBO) in the standard solution
 M_1 = Mass in 'g' pipernoyl butoxide (PBO) in the standard solution
 M_2 = Mass in 'g' of sample taken for test
 P = Percent purity of pipernoyl butoxide (PBO) standard

$$\text{Pipernoyl butoxide content in aerosol, \% by mass} = \frac{\% \text{ A.I in premix} \times \text{Weight of premix (W}_6\text{)}}{\text{Weight of Aerosol (W}_5\text{)}}$$

Sl. No.	Name of test	Result	Unit	Method of Analysis
1.	Active ingredient		%	
2.				

Remark / Reference :

Analyzed by	Name	
	Dated signature	
Checked by	Name	
	Dated signature	

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