 FC-PF-263

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

Sr. No. in Scope NABL / NON NABL

Flow Chart for Analysis of Tricyclazole Content in Formulation Sample

Date of Analysis	

S. No.	Step	Execution		Executed by
		R1	R2	
1.	Sample No.			
2.	Name of Sample			
3.	**Procedure**			
3.1.	**Preparation of Mobile Phase**			
3.1.1	Pipette out 10mL of 85% orthophosphoric acid (HPLC grade) in a 100 mL volumetric flask and make up to the mark with HPLC water.			
3.1.2.	Adjust the pH of HPLC water with 10% Ortho phosphoric acid (3.1.1) to pH 2.85			
3.1.3.	Mix acetonitrile and pH adjusted water (3.1.2) in the proportion of 50:50 (v/v)			
3.1.4.	Pass through the 0.45 µm membrane filter under vaccum.			
3.1.5.	Sonicate the mixture to Homogenize.			
3.1.6	Allow to attain room temperature.			
3.2	**Preparation of Internal Standard Solution**			
3.2.1	Weigh 1.5 g of Acetophenone in 100 ml volumetric flask	g	g	
3.2.2	*Note down the serial No. of the balance log book*			
3.2.3	Add 50 ml of Acetonitrile, mix & sonicate for 5 minutes. Cool to room temperature & make up to the mark with acetonitrile.			
3.2.4	Stopper and shake well to homogenize.			
3.3	**Preparation of standard solution**			
3.3.1	Note the purity of the standard	%	%	
3.3.2	Weigh 50 mg a. i. of Standard in a 100 ml volumetric flask	g	g	
3.3.3	*Note down the serial No. of the balance log book*			
3.3.4	Add 50 mL methanol, sonicate for 5 min to dissolve the material.			
3.3.5	Add 5 ml of internal standard solution (3.2.4)	mL	mL	
3.3.6	Dilute up to the mark with methanol and mix well (Stock A).			

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3.3.7	Pipette out 1 mL of Stock A (3.3.6) into a 100 mL volumetric flask			
3.3.8	Dilute up to the mark with acetonitrile. Stopper and shake well to homogenize.			
3.4	**Preparation of sample solution**			
3.4.1	Note down the percent active ingredient declared on the sample	%	%	
3.4.2	Weigh 50 mg a. i. of sample in a 100 ml volumetric flask	g	g	
3.4.3	*Note down the serial No. of the balance log book*			
3.4.4	Add 50 mL methanol, sonicate for 5 min to dissolve the material.	ml	ml	
3.4.5	Add 5 ml of internal standard solution (3.2.4)			
3.4.6	Dilute up to the mark with methanol and mix well (Stock B).			
3.4.7	Pipette out 1 mL of Stock B (3.4.6) into a 100 mL volumetric flask			
3.4.8	Dilute up to the mark with acetonitrile. Stopper and shake well to homogenize.			
3.4.9	Filter the sample solution through 0.45µ membrane filter			
4.	**HPLC Parameters**			
4.1	**Column**			
4.1.1	C_{18}, Particle Size: 5µ			
4.1.2	Length: 250 mm			
4.1.3	I.D.: 4.6 mm			
4.2	**Mobile Phase**			
4.2.1	Acetonitrile : Water, pH adjusted to 2.85 (50:50)			
4.2.2	Flow Rate : 0.85 ml/min			
4.3	**Detector:** UV			
4.4	**Wave Length**: 231 nm			
4.5	**Injection Volume:** 20 µl			
5.	**Result**			
	Sample chromatogram no.			
	Standard chromatogram no.			

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6. Calculation:

Tricyclazole content, % by mass =

$$\frac{A_2 \times A_3 \times M_1}{A_1 \times A_4 \times M_2} \times P$$

Where,

M_1 =Mass in 'g' of Tricyclazole standard
M_2 = Mass in 'g' of sample taken for test
A_1 = Peak area of Tricyclazole in the standard solution
A_2 = Peak area of Tricyclazole in the sample solution
A_3 = Peak area of internal standard in the standard solution
A_4 = Peak area of internal standard in the sample solution
P = Percent purity of Tricyclazole standard

Result:

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