

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

Sr. No. in Scope

NABL / NON NABL

Flow Chart for Analysis of Clodinafop- Propargyl 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 1.35 mL of orthophosphoric acid (HPLC grade) in to 1000 mL HPLC water and mix well by sonication.			
3.1.2.	Pass through the 0.45 µm membrane filter under vaccum.			
3.1.3.	Mix acetonitrile and acidified water (3.1.2) in the proportion of 70 : 30 (v/v). Sonicate the mixture to Homogenize. Allow to attain room temperature.			
3.2	Preparation of standard solution			
3.2.1	Note the purity of the standard	%	%	
3.2.2	Weigh 50 mg a. i. of Standard in a 50 ml volumetric flask	g	g	
3.2.3	<i>Note down the serial No. of the balance log book</i>			
3.2.4	Add 20 mL mobile phase (3.1.3), sonicate for 5 min to dissolve the material.			
3.2.5	Dilute up to the mark with mobile phase (3.1.3)(Stock A).			
3.2.6	Pipette out 5 mL of Stock A (3.2.5) into a 25 mL volumetric flask			
3.2.7	Dilute up to the mark with mobile phase (3.1.3). Stopper and shake well to homogenize.			
3.3	Preparation of sample solution			
3.3.1	Note down the percent active ingredient declared on the sample	%	%	
3.3.2	Weigh 50 mg a. i. of sample in a 50 ml volumetric flask	g	g	
3.3.3	<i>Note down the serial No. of the balance log book</i>			
3.3.4	Add 20 mL mobile phase (3.1.3), sonicate for 5 min to dissolve the material.	ml	ml	
3.3.5	Dilute up to the mark with mobile phase (3.1.3) and mix			

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Revision No.	:	00	Issue Date	:	15/09/2014
Revision Date	:	--	Next Revision Date	:	15/09/2016
Prepared By		Checked By		Approved by	Issued By
Ms. T. Sridevi (Deputy Quality Manager)		Dr. Nirmali Saikia (Technical Manager)		Dr. Abhay Ekbote (Director PM)	Mr. C.V. Rao (Quality Manager)

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	well (Stock B).			
3.3.6	Pipette out 5 mL of Stock B (3.3.5) into a 25 mL volumetric flask			
3.3.7	Dilute up to the mark with mobile phase(3.1.3). Stopper and shake well to homogenize.			
3.3.8	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: Phosphoric acid (70:30:0.04)(as per 3.1.3)			
4.2.2	Flow Rate : 1.40 ml/min			
4.3	Detector: UV			
4.4	Wave Length: 225 nm			
4.5	Injection Volume: 20 µl			
5.	Data			
	Sample chromatogram no.			
	Standard chromatogram no.			

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6. Calculation:

Clodinafop-propargyl content, % by mass, $= \frac{M_1 \times A_2}{M_2 \times A_1} \times P$	Where, M_1 = Mass in 'g' of standard clodinafop propargyl M_2 = Mass in 'g' of sample taken for the test A_2 = Peak area of clodinafop propargyl in the sample solution A_1 = Peak area of clodinafop propargyl in the standard solution P = Percent purity of standard clodinafop propargyl
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Result:

Sl.No.	Name of test	Result	Unit	Method of Analysis
1.	Active ingredient		%	In house method (SOP/PFRAC/249)
Remark / Reference :				

Analyzed by	Name	
	Dated signature	
Checked by	Name	
	Dated signature	

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