

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

Sr. No. in Scope

NABL / NON NABL

**Flow chart for analysis of Chlorpyrifos and Cypermethrin in formulation sample**

Sl. No.	Step	Date of Analysis	
		Execution	Executed By
1.	Sample No.		
2.	Name of Sample		
3.	<b>Procedure</b>		
<b>3.1</b>	<b>Preparation of Internal Standard</b>		
3.1.1	Weigh 0.5 g Dioctyl phthalate (DOP) taken into 250 ml volumetric flask	g	
3.1.2	<i>Note down the S.No. of balance log book.</i>		
3.1.3	Dissolve it and make the volume up to the mark with ethyl acetate		
<b>3.2</b>	<b>Preparation of Standard Cypermethrin Solution</b>		
3.2.1	Purity of standard Cypermethrin	%	
3.2.2	Weigh 0.15 g a.i of standard into a 50 ml volumetric flask.	g	
3.2.3	<i>Note down the S.No. of balance log book.</i>		
3.2.4	Disolve and dilute up to the mark with ethyl acetate (Stock A)		
<b>3.3.</b>	<b>Preparation of mixture of Chlorpyrifos and Cypermethrin Standard Solution:</b>		
3.3.1	Purity of standard Chlorpyrifos	%	
3.3.2	Weigh 0.15 g a.i. of chlorpyrifos standard into a 50 ml volumetric flask	g	
3.3.3	<i>Note down the S.No. of balance log book.</i>		
3.3.4	Add 5 mL of stock A (3.2.4) and 25 mL of internal standard solution (3.1.3)		
3.3.5	Dilute up to the mark with ethyl acetate and mix well.		
<b>3.4</b>	<b>Preparation of Sample</b>		
3.4.1	Note down the percent active ingredient content of chlorpyrifos and cypermethrin declared on the sample, respectively.	% %	
3.4.2	Weigh 0.3 g of the sample into a 50 ml volumetric flask	g	
3.4.3	<i>Note down the S.No. of balance log book.</i>		
3.4.4	Add 25 mL of internal standard solution (3.1.3)	ml	
3.4.5	Dilute up to the mark with ethyl acetate and mix well.		

Name of the Laboratory :		<b>Pesticide Formulation &amp; Residue Analytical Centre, PMD, NIPHM, Hyderabad</b>	
Document No.	:	FC-PF-206	Document Name : Flow chart for analysis of Chlorpyrifos & Cypermethrin, content, % by mass
Revision No.	:	01	Issue Date : 01/07/2011
Revision Date	:	1/12/2013	Next Revision Date : 01/12/2015
Prepared By		Checked By	Approved & Issued By
Mrs. T. Sridevi (Deputy Technical Manager)		Mr. C.V. Rao (Technical Manager)	Dr. Abhay Ekbote (Director PM & Quality Manager)

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

<b>4.</b>	<b>GC Parameters</b>		
<b>4.1</b>	<b>Column:</b> 3 % OV -101 on Gas chrom Q (80-100) mesh		
4.1.1	Length: 100 cm		
4.1.2	I.D: 2 mm		
<b>4.2</b>	<b>Gas</b>		
4.2.1	Carrier: Nitrogen: 30 ml/min		
4.2.2	Hydrogen: 45 ml/min		
4.2.3	Air: 450 ml/min		
<b>4.3</b>	<b>Temperature</b>		
4.3.1	Oven: 180°C for 2.5 min @ 15°C/min upto 240°C for 2.5 min		
4.3.2	Injector: 250°C		
4.3.3	Detector: 270°C		
<b>4.4</b>	<b>Injection volume:</b> 1 µl		
<b>5.</b>	<b>Result</b>		
	Sample chromatogram no.		
	Standard chromatogram no.		

**6. CALCULATION:**

**Chlorpyrifos 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<sub>1</sub>= Peak area of chlorpyrifos in the sample solution

A'IS'<sub>1</sub>= Peak area of internal standard in the sample solution

A'IS'<sub>2</sub>= Peak area of internal standard in the standard solution

A<sub>2</sub>= Peak area of chlorpyrifos in the standard solution

M<sub>1</sub>= Mass in 'g' of chlorpyrifos in the standard solution

M<sub>2</sub>= Mass in 'g' of sample taken for test

P = Percent purity of chlorpyrifos standard

Name of the Laboratory :		<b>Pesticide Formulation &amp; Residue Analytical Centre, PMD, NIPHM, Hyderabad</b>	
Document No.	:	FC-PF-206	Document Name : Flow chart for analysis of Chlorpyrifos & Cypermethrin, content, % by mass
Revision No.	:	01	Issue Date : 01/07/2011
Revision Date	:	1/12/2013	Next Revision Date : 01/12/2015
Prepared By		Checked By	Approved & Issued By
Mrs. T. Sridevi (Deputy Technical Manager)		Mr. C.V. Rao (Technical Manager)	Dr. Abhay Ekbote (Director PM & Quality Manager)

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

**Cypermethrin content, % by mass =**

$$\frac{A_3 \times A'IS'_2 \times M}{A'IS'_1 \times A_4 \times M_2} \times P$$

**Where,**

A<sub>3</sub>= Peak area of cypermethrin in the sample solution

A'IS'<sub>1</sub>= Peak area of internal standard in the sample solution

A'IS'<sub>2</sub>= Peak area of internal standard in the standard solution

A<sub>4</sub>= Peak area of cypermethrin in the standard solution

P = Percent purity of cypermethrin standard

M<sub>2</sub>= Mass in 'g' of sample taken for test

M= Mass in 'g' of cypermethrin in standard.

**Result:**

Sl. No.	Name of Test	Result	Unit	Method of Analysis
1.	Active ingredient (Chlorpyrifos)		%	(IS - 15235 : 2002)
2.	Active ingredient (Cypermethrin)		%	(Reaffirmed 2009)
Remark / Reference :				

Analyzed by	Name	
	Dated Signature	
Checked by	Name	
	Dated Signature	

Name of the Laboratory :		<b>Pesticide Formulation &amp; Residue Analytical Centre, PMD, NIPHM, Hyderabad</b>			
Document No.	:	FC-PF-206	Document Name	:	Flow chart for analysis of Chlorpyrifos & Cypermethrin, content, % by mass
Revision No.	:	01	Issue Date	:	01/07/2011
Revision Date	:	1/12/2013	Next Revision Date	:	01/12/2015
Prepared By		Checked By		Approved & Issued By	
Mrs. T. Sridevi (Deputy Technical Manager)		Mr. C.V. Rao (Technical Manager)		Dr. Abhay Ekbote (Director PM & Quality Manager)	