

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

Sr. No. in Scope

NABL / NON NABL

Flow chart for analysis of Dicofol in formulation

Date of Analysis

Sl. No.	Step	Execution		Executed By
1.	Sample No.			
2.	Name of Sample			
3.	Preparation of Standard solutions (0.1 N):	R₁	R₂	
3.1	Weigh 16.9 g of AgNO ₃ and dissolve in water to get 1L solution	g	g	
3.2	Weigh 10.5 g of KSCN and dissolve in water to get 1L solution.	g	g	
4.	Standardization of AgNO₃:			
4.1	Weigh 0.1-0.15 g NaCl or KCl GR/AR grade in duplicate into a 250 mL conical flask.	g	g	
4.2.	Dissolve in 50-70 mL of water			
4.3	Add Potassium chromate indicator (5% aqueous solution).			
4.4	Titrate with AgNO ₃ solution taken in burette slowly with continuous stirring to pale reddish brown end point.			
4.5	Titre value for the 2 replications	mL	mL	
5.	Standardization of KSCN:			
5.1.	Pipette out 25 mL of standardized 0.1 N AgNO ₃ solution into a 250 mL conical flask in duplicate.			
5.2.	Add 10 mL of 6 N Nitric acid			
5.3.	Add 1 mL of Ferric alum indicator			
5.4.	Titrate with 0.1 N KSCN taken in burette slowly with continuous stirring to pale reddish brown end point.			
5.5.	Titre value for the 2 replications	mL	mL	
6.	Procedure			
6.1	Preparation of Sample Solution			
6.1.1.	Percent active ingredient declared on the sample	%	%	
6.1.2	Weigh Sample equivalent to 0.5 g of active ingredient, transfer to 500ml GG flat bottom flask, quantitatively with 50 mL of ethanol	g	g	
6.1.3	Add KOH pellets (1.4 g) to get 50 mL of 0.5 N ethanol			
6.1.4	Place the flask on heating mantle and connect the neck of the flask to water cooled condenser and boil the solution under gentle reflux for 90 min.			
6.1.5	At the end of the time stop heating and allow the flask to cool slightly, rinse the condenser with 25 ml of 95% ethanol.			

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Revision No.	:	01	Issue Date	:	01/07/2013
Revision Date	:	26/03/2014	Next Revision Date	:	26/03/2016
Prepared By		Checked By		Approved & Issued By	
Mrs. T. Sridevi (Assistant Scientific Officer)		Mr. C.V. Rao (Technical Manager)		Dr. Abhay Ekbote (Director PM) & Quality Manager	

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6.1.6.	Add 2-3 drops phenolphthalein indicator solution, pink colour appears.			
6.1.7.	Add sufficient 1:1 Nitric acid solution to turn the solution colourless.			
6.1.8.	Add an additional 5 ml of 1:1 Nitric acid and 50ml of the standard silver nitrate solution through pipette or burette. Thoroughly mix the contents of the conical flask.			
6.1.9.	Add 5ml of nitrobenzene to the flask, stir vigorously and add 1 mL of ferric alum indicator.			
6.1.10	Titrate the excess of silver nitrate with the standard potassium thiocyanate solution with continuous stirring, until the appearance of faint but permanent brick red end point.			
6.1.11	Titre value for sample is	mL	mL	
6.2.	Reagent Blank			
6.2.1.	Follow the steps from 6.1.2 to 6.1.10, taking all the reagents except sample for reagent blank.			
6.2.2	Titre value for reagent blank is	mL	mL	

7. Calculation:

$\text{Normality of AgNO}_3 = \frac{\text{Wt. of NaCl} \times 1000}{\text{Volume of AgNO}_3 \text{ consumed} \times 58.46}$
$\text{Normality of KSCN} = \frac{\text{Normality of AgNO}_3 \times \text{volume of AgNO}_3 \text{ taken}}{\text{volume of KSCN consumed}}$

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(mL of KSCN consumed for Blank)-(mL of KSCN consumed for sample) XN X12.351

Active ingredient content, = -----

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% by mass Weight of sample (g)

Where,
N= Normality of KSCN solution

8. Result:

Sl. No.	Name of test	Result	Unit	Method of Analysis
1.	Active ingredient		% (w/w)	IS: 5278-1969

Remark / Reference :

Analyzed by	Name	
	Dated signature	
Checked by	Name	
	Dated signature	

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