

Flow Chart for Analysis of Sulphur in formulation

Date of Analysis

Sl. No.	Step	Execution		Executed By
1.	Sample No.			
2.	Name of Sample			
2.1	Sample Description			
3.	Preparation of Standard solutions (0.1 N):	R₁	R₂	
3.1	Weigh 24.8 g of Sodium thiosulphate and dissolve in water to get 1 L solution	g	g	
3.2	Note the Serial No. in the balance logbook			
4.	Standardization of Sodium thiosulphate:			
4.1	Weigh 0.07-0.08 g of KIO ₃ in a conical flask			
4.2	Note the Serial No. in the balance logbook			
4.3	Add 40-50 ml of water and dissolve			
4.4	Add 2 g of KI			
4.5	Note the Serial No. in the balance logbook			
4.6	Add 2 ml of 5N H ₂ SO ₄			
4.7	Titrate Immediately with Sodium thiosulphate solution in burette till pale yellow appears			
4.8	Add 1 ml of starch indicator (Color of solution changes to blue)			
4.9	Then again titrate with Na ₂ S ₂ O ₃ till it changes to colorless (endpoint)			
4.10	Titre value is			
5.	Preparation of Iodine solution			
5.1	Weigh 30-40 g of KI into an Iodine flask	g	g	
5.2	Note the Serial No. in the balance logbook			
5.3	Add 50-60 ml of water and dissolve			
5.4	Add 13 g of Iodine to 5.3 and dissolve thoroughly	g	g	
5.5	Note the Serial No. in the balance logbook			
5.6	Transfer into a amber colored bottle (Keep it for overnight)			
6.	Standardization of Iodine solution			
6.1	Take 25 ml of standardized Iodine solution in a conical flask			
6.2	Add 1 ml of starch solution			

Name of the Laboratory : Pesticide Formulation & Residue Analytical Centre, PMD, NIPHM, Hyderabad					
Document No.	:	FC-PF-245	Document Name	:	Flow chart for analysis of Sulphur in Formulation
Revision No.	:	01	Issue Date	:	15/07/2013
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)	

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

6.3	Titrate against sodium thiosulphate solution, end point will be colorless to blue			
6.4	Titre value is			
7.	Procedure			
7.1	Weigh accurately 0.1 g of sample (0.125 g of 80% WP) and transfer into a 500 ml GG flat bottom flask			
7.2	Add 10 ml alcohol to dissolve sulphur (If the formulation is DP)			
7.3	Add 30-40 ml of water			
7.4	Add 2 g Sodium sulphite			
7.5	Add liquid paraffin and few glass beads			
7.6	Fix the flask to the water cooled condenser & keep for reflux for 40 minutes			
7.7	Cool the flask, rinse the condenser with water and remove the flask			
7.8	Add 10 ml of formaldehyde			
7.9	Add 10 ml of Carbon tetrachloride			
7.10	Add 10 ml of Acetic acid			
7.11	Titrate immediately with standard Iodine solution using freshly prepared starch solution as indicator			
7.12	End point will be the first appearance of blue color throughout the solution			
7.13	Follow the same procedure from 7.2 to 7.12 for reagent blank			
7.14	Titre value for sample is			
7.15	Titre value for blank is			

6. Calculation:

$$\text{Normality of Na}_2\text{S}_2\text{O}_3 = \frac{\text{Wt. of KIO}_3 \times 1000}{\text{Volume of Na}_2\text{S}_2\text{O}_3 \text{ consumed} \times 35.67}$$

Name of the Laboratory : Pesticide Formulation & Residue Analytical Centre, PMD, NIPHM, Hyderabad					
Document No.	:	FC-PF-245	Document Name	:	Flow chart for analysis of Sulphur in Formulation
Revision No.	:	01	Issue Date	:	15/07/2013
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)	

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

$$\text{Normality of Iodine} = \frac{\text{Normality of Na}_2\text{S}_2\text{O}_3 \times \text{volume of Na}_2\text{S}_2\text{O}_3 \text{ taken}}{\text{Volume of Iodine consumed}}$$

$$\text{Active ingredient content, \% by mass} = \frac{(\text{mL of Iodine for sample}) - (\text{mL of Iodine for blank}) \times N \times 3.206}{\text{Weight of sample (g)}}$$

Where N = Normality of Iodine solution

Result:

Sl. No.	Name of Test	Result	Unit	Method of Analysis
1.	Active ingredient (Sulfur)		%	IS : 6444 - 1979 (Reaffirmed 2002)
Reference in daily workbook:				

Analyzed by	Name	
	Dated signature	
Checked by	Name	
	Dated signature	

Name of the Laboratory : Pesticide Formulation & Residue Analytical Centre, PMD, NIPHM, Hyderabad					
Document No.	:	FC-PF-245	Document Name	:	Flow chart for analysis of Sulphur in Formulation
Revision No.	:	01	Issue Date	:	15/07/2013
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)	