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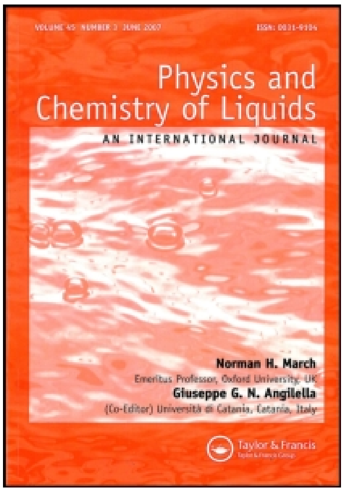

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A SPECTROPHOTOMETRIC METHOD FOR THE DETERMINATION OF PROPOXUR

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A simple spectrophotometric technique is reported for the determination of propoxur using the coupling complexing agents namely 2-amino, 4-nitrophenol and 2,4-dimethoxy aniline. The $\lambda_{\max}$ observed for these coloured complexes were at 402 and 460 nm respectively. The recoveries of propoxur pesticides obtained using the above procedure were reported in their formulations, water and food grains.

Keywords: Propoxur; 2-amino; 4-nitrophenol; 2,4-dimethoxy aniline; Spectrophotometric method

INTRODUCTION

Propoxur, a non-systemic insecticide belonging to the carbamate class of pesticides, has its potential use in controlling household pests like ants, bugs, flies and mosquitos, and demands a simple, reliable and precise procedure for its estimation in various environments like waters, vegetable matters and food grains.

Colorimetric [1–5], Chromatographic [6–9], Polarographic [10] and Spectrophotometric [11–17] methods were employed for the determination of propoxur in various environments like plant and animal tissues, vegetables, fruits, food grains, different types water and milk samples. The present study reports a simple, rapid and convenient spectrophotometric procedure for the determination of propoxur in their formulations, waters and certain food grains.

EXPERIMENTAL

The analytical grade of propoxur pesticide samples in the form of 1% Baygon spray, 2% Baygon bait, 4% Baygon and 70% wettable powder of Bayer India Ltd., India

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were used for the present study. The solvents like methanol, chloroform and acetone were purified and employed for the present study.

The reagents like sodium hydroxide (2%), sodium nitrite (0.3%), potassium permanganate solution, potassium carbonate (0.1%) solutions, 2-amino, 4-nitrophenol (0.2%) and 2,4-dimethoxy aniline (0.2%) solutions were employed for the present study. The spectral data in the UV–Vis region for the coupled complexes were recorded on a Shimadzu (model-240) recording double beam spectrophotometer and reported in Table I.

The complexes of propoxur were prepared by azo coupling reaction technique. Propoxur samples were hydrolysed using 2% aqueous NaOH to yield their corresponding phenols. Diazonium salts of 2-amino, 4-nitrophenol and 2,4-dimethoxy aniline were prepared using 0.3% $NaNO_2$ and 1N HCl for coupling reaction. The pH of the complexes was maintained between 11 and 12. The spectra for the above complexes were recorded in the UV–Vis regions and was reported in the Table I. Beer's law was obeyed over the concentration range of 0.4–10 and 0.4–12 ppm respectively in these complexes. This data was used for the analysis of the samples for estimation.

Determination of Propoxur in its Formulations

Propoxur in 1% Baygon spray, 2% Baygon bait, 4% Baygon and 70% Wettable powder were analysed using the aforesaid procedure by coupling diazotised compounds of 2-amino, 4-nitrophenol and 2,4-dimethoxy aniline [18,19] which are presented in the Tables II and III.

Determination of Propoxur in Water Samples

Distilled and tap water samples were fortified with the concentrations ranging from 0.6 to 4.8 ppm in methanol. The fortified water samples were extracted with chloroform. The combined extracts were washed with 0.1 M potassium carbonate solution to break any emulsion formed during the extraction and dried over anhydrous sodium sulphate. Finally chloroform was evaporated to dryness on a steam bath and the residue was dissolved in methanol and the amount was determined.

TABLE I Optical characteristics, precision and accuracy of the method using 2-amino, 4-nitrophenol and 2,4-dimethoxy aniline as coupling agents

S. No.	*Parameter*	*2-amino, 4-nitrophenol*	*2,4-dimethoxy aniline*
1	Concentration range	0.4–10.0 $\mu g\,mL^{-1}$	0.4–12.0 $\mu g\,mL^{-1}$
2	λ_{max}	402 nm	460 nm
3	Stability of the colour	72 h	68 h
4	Molar absorptivity	$3.012 \times 10^4\,L\,mol^{-1}\,cm^{-1}$	$2.970 \times 10^4\,L\,mol^{-1}\,cm^{-1}$
5	Sandell's sensitivity	0.0104 $\mu g\,cm^{-2}$	0.0211 $\mu g\,cm^{-2}$
6	*Relative standard deviation (RSD %)	0.98	0.30
7	Correlation coefficient	1.0040	0.9986
8	Relative error (%)	0.80	0.20

*Calculated for ten samples containing same amount of carbamate.

TABLE II Determination of propoxur in insecticidal formulations using diazotised 2-amino, 4-nitrophenol as a coupling agent

Labelled amount sample No.	*1% Baygon spray*	*2% Baygon bait*	*4% Baygon*	*70% Wettable powder*
1	0.94	1.92	3.84	66.03
2	0.94	1.92	3.84	65.88
3	0.96	1.91	3.73	65.43
4	0.96	1.88	3.75	67.14
5	0.96	1.88	3.76	65.80
6	0.96	1.92	3.76	65.96
7	0.96	1.89	3.85	66.18
8	0.96	1.89	3.78	66.31
Av.	0.95	1.90	3.79	66.09
SD	0.009	0.019	0.047	0.499

TABLE III Determination of propoxur in insecticidal formulations using diazotised 2,4-dimethoxyaniline as a coupling agent

Labelled amount sample No.	*1% Baygon spray*	*2% Baygon bait*	*4% Baygon*	*70% Wettable powder*
1	0.95	1.85	3.80	65.00
2	0.95	1.86	3.81	65.20
3	0.93	1.82	3.82	65.30
4	0.94	1.87	3.91	67.00
5	0.94	1.88	3.84	65.80
6	0.96	1.88	3.92	67.30
7	0.96	1.89	3.78	64.90
8	0.95	1.90	3.86	66.50
Av.	0.95	1.87	3.84	65.88
SD	0.011	0.025	0.051	0.941

Determination of Propoxur in Grain Samples (Rice and Wheat)

The grain samples (rice and wheat) of 50 gm each were taken in warming blender and blended for 5 min. with 100 mL of chloroform. The samples were fortified with different concentrations of insecticides in methanol and blended for 3 min. Chloroform was filtered and the residue as retained. The residue was washed twice with 10 mL of chloroform and blended for 2 min. The chloroform extracts were evaporated on a steam bath and the residue was dissolved in methanol and the amount was determined by the procedure outlined earlier.

Results and Discussion

The characteristics of the spectrophotometric methods summarised in Table I reveal that 2-amino, 4-nitorphenol, 2,4-dimethoxy aniline can be successfully employed as coupling agents for the determination of propoxur. The molar absorptivity of the coloured complexes formed are in the range of 2.970×10^4 and $3.012 \times 10^4\,\mathrm{L\,mol^{-1}\,cm^{-1}}$. These values show that the sensitivity of the method is high. The above inference is substantiated by the low Sandell's sensitivity values.

The values of correlation coefficient are very close to unity. This indicates that the absorbance depends on the concentration of the insecticide. The additional advantage of the methods are instantaneous formation of coloured complexes at room temperature and remain stable for longer period when compared to the recoveries reported by Naidu and Naidu [14]. The relative standard deviation values and percentage error values indicate that the new procedures offer good precision and accuracy.

The data included in Tables II and III show that the active ingredient present in the formulations of propoxur can be successfully determined spectrophotometrically using the new reagents. The recoveries are favourably compared to the recoveries reported by Naidu and Naidu [14]. The amount of propoxur determined experimentally is very close to the values of their formulations and are given in Tables II and III. This observation suggests that the other ingredients present in the formulations do not interfere. Hence, the aforesaid reagents can be successfully adopted for a routine checkup of the purity of the commercial insecticidal formulations.

The data given in Tables IV and V suggest that the percentage of recovery from fortified tap and water samples are in the ranges of 95.2–98.33 and 93.75–96.66 respectively. The percentage ranges of recoveries of the pesticides in rice and wheat samples are in the ranges of 94.7–96.66 and 97.40–98.85 respectively (Tables VI and VII). It appears in all the cases, the percentage of recoveries are highest and therefore the present technique is a simple, rapid and sensitive to analyse the pesticide samples.

These methods do not involve elaborate clean up procedures required by other methods. Hence, the methods described here would serve as an additional techniques for the determination of propoxur in fortified water samples and food grains. It is

TABLE IV Recovery of propoxur from fortified water samples using diazotised 2-amino, 4-nitrophenol as a coupling agent

Sample No.	*Fortification level* (ppm)	*Tap water*		*Distilled water*	
		Amt. Rec. (ppm)	*Rec.* %	*Amt. Rec.* (ppm)	*Rec.* %
1	0.8	0.77	96.00	0.75	94.00
2	1.6	1.57	98.06	1.54	96.18
3	2.4	2.31	96.25	2.27	94.58
4	3.2	3.08	96.25	3.03	94.68
5	4.0	3.87	96.75	3.81	95.15
6	4.8	4.57	95.20	4.50	93.75

TABLE V Recovery of propoxur from fortified water samples using diazotised 2,4-dimethoxy aniline as a coupling agent

Sample No.	*Fortification level* (ppm)	*Tap water*		*Distilled water*	
		Amt. Rec. (ppm)	*Rec.* %	*Amt. Rec.* (ppm)	*Rec.* %
1	0.6	0.59	97.50	0.57	95.33
2	1.2	1.17	97.83	1.15	95.58
3	1.8	1.76	97.77	1.73	95.83
4	2.4	2.36	98.12	2.31	96.25
5	3.0	2.94	98.00	2.89	96.33
6	3.6	3.54	98.33	3.48	96.66

TABLE VI Recovery of propoxur from fortified grains using diazotised 2-amino, 4-nitrophenol as coupling agent

Sample No.	*Fortification level* (ppm)	*Rice*		*Wheat*	
		Amt. Rec. (ppm)	*Rec.* %	*Amt. Rec.* (ppm)	*Rec.* %
1	0.8	0.76	94.70	0.78	97.40
2	1.6	1.53	95.40	1.56	97.77
3	2.4	2.30	96.00	2.35	98.03
4	3.2	3.09	96.40	3.14	98.24
5	4.0	3.84	96.00	3.90	97.50
6	4.8	4.62	96.25	4.71	98.12

TABLE VII Recovery of propoxur from fortified grains using diazotised 2,4-dimethoxy aniline as coupling agent

Sample No.	*Fortification level* (ppm)	*Rice*		*Wheat*	
		Amt. Rec. (ppm)	*Rec.* %	*Amt. Rec.* (ppm)	*Rec.* %
1	0.6	0.58	96.25	0.59	97.50
2	1.2	1.15	96.66	1.18	98.85
3	1.8	1.73	95.83	1.77	98.12
4	2.4	2.32	96.91	2.36	98.40
5	3.0	2.86	95.30	2.95	98.33
6	3.6	3.48	96.66	3.55	98.52

also worthwhile to note that the holding tendencies of the propoxur pesticide in various environments under study are as follows:

$$\text{Distilled water} > \text{Tap water}$$

$$\text{Rice} > \text{Wheat}$$

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