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NOTE

Spectrophotometric Determination of Carbosulfan in Various Environments using Oxidative Coupling

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A method for the determination of carbosulfan in environmental samples using oxidative coupling is being described. The method comprises of alkaline hydrolysis of the pesticide and the resulting phenol is reacted with 2,4-dimethoxy aniline in the presence of acidified $K_2Cr_2O_7$. The dye product formed is extracted into $CHCl_3$ and the absorbance measured at 430 nm.

Key Words: Carbosulfan, Environmnetal samples, Oxidative coupling, Sectrophotometry.

The pesticidal properties of carbosulfan, a member of carbamate family, were reported in literature[1,2]. Bruce *et al.*[3] studied the pesticidal properties if carbosulfan in water, soil and plants. The presence of pesticides were estimated in water and food grains using colorimetic technique[4,5]. These methods were based on alkaline hydrolysis of the pesticide and coupling the phenolic product with various diazo compounds and the subsequent determination was in aqueous medium. The methods have their inherent drawback due to lower sensitivity and also due to the reaction of the diazo reagent with other species present in the medium resulting in the instability of the coloured compound. The phenolic product formed by alkaline hydrolysis with 2,4-dimethoxy aniline in the presence of acidified potassium dichromate to yield coloured cyanogen compound. The method developed is applied for the determination of the pesticide in environmental samples.

25 % Seed treat and 91.4 % technical grade samples of carbosulfan supplied by M/s Rallis Inida Ltd., Bangalore were used in the present work. Carbofuran is generated by the reaction of carbosulfan with 0.1 mL of 2 N H_2SO_4 and NaOH is used to hydrolyze carbofuran to yield corresponding phenol.

3 g $K_2Cr_2O_7$ was dissolved in 100 mL distilled water, 4 N H_2SO_4 was used in the course of the experiments. 2 g of 2,4-dimethoxy aniline dissolved in distilled water and diluted to 100 mL. Shimadzu UV-240 recording spectrophotometer was employed for the absorbance measurements.

Procedure: 20 mL of insecticide solution was taken in a 100 mL beaker to which 5 mL of 2 % NaOH was added and allowed to stand for 5 min for complete hydrolysis. The pH of the resulting solution is adjusted to 3.5 by sulphuric acid and ammonia solution. The solution was transferred into a 50 mL separating funnel and allowed to stand for 2 min and then 3 mL of 2,4-dimethoxy aniline was added followed by 3 mL of potassium dichromate solution and equilibrated. The orange coloured dye formed was extracted into 10 mL chloroform. The absorbance of the chloroform extract was measured at 472 nm against a reagent blank.

Formulations: Samples of formulations, equivalent to about 100 µg of the active insecticide were treated with 25 mL methanol and the supernatant solution was separated by decantation into a 100 mL standard flask. The residue was repeatedly washed with 10 mL portion of methanol. The combined methanol extracts were made up to 100 mL. Analysis was carried out as described.

Water and food grain samples: Distilled and tap water, and food grains, wheat and rice samples were fortified with 0.5-0.3 ppm of carbosulfan. These fortified samples were extracted independently with chloroform and the residues were dissolved in methanol and analyzed.

Carbosulfan on alkaline hydrolysis produces a phenolic compound, which on coupling with 2,4-dimethoxy aniline in presence of oxidizing agent forms a coloured cyanogen compound. The cyanogen compound extracted at pH 3.5, into chloroform exhibits absorbance maximum at 430 nm.

The reaction of the phenolic compound with 2,4-dimethoxy aniline was studied in the pH range 1-6 and the absorbance of the coloured cyanogen was maximum at pH 3.5. Among the solvents, C_6H_6, CCl_4 and $CHCl_3$, extraction of the coloured dye was maximum into $CHCl_3$. Further, the dye solution was stable in this solvents for more than 2 d. Beer's law was obeyed over the concentration range 0.1-1.0 ppm of the pesticide. The coloured dye has a molar absorptivity of 3.09×10^5 L mol^{-1} cm^{-1}. The absorption data of the dye is presented in Table-1.

TABLE-1
DETERMINATION OF CARBOFURAN BY OXIDATIVE COUPLING WITH 2,4-DIMETHOXY ANILINE

Concentration range (ppm)	0.1-1.0
λ_{max} (nm)	430
Colour stability (h)	> 24
Molar absorbivity (L/mol cm)	3.09×10^5
Sandell's sensitivity (µg/cm^2)	0.0235
Relative standard deviation (10 samples)	0.61
Correlation coefficient	0.9998
Relative error	0.48

Data relating to the analysis of the pesticide in 25 % seed treat and 91.4 % technical formulations, 8 samples each is presented in Table-2.

TABLE-2
DETERMINATION OF CARBOSULFAN-
INSECTICIDAL FORMULATIONS

Sample number	Labeled amount	
	25 % seed treat	91.4 Technical
1	24.76	89.20
2	24.87	89.59
3	24.75	90.56
4	24.81	98.98
5	24.87	90.76
6	24.86	90.59
7	24.56	90.78
8	24.63	90.45
Average	24.56	90.24
Standard deviation	0.12	0.58

TABLE-3
RECOVERY OF CARBOSULFAN FROM FORTIFIED
WATER SAMPLES AND GRAINS

Sample no.	Fortification level (ppm)	Water samples				Grains			
		Tap water		Distilled water		Rice		Wheat	
		Amount (ppm)	%	Amount (ppm)	%	Amount (ppm)	%	Amount (ppm)	%
1	0.8	0.77	95.75	0.76	95.00	0.75	93.25	0.76	95.00
2	1.6	1.54	96.26	1.52	95.00	1.50	95.77	1.53	95.75
3	2.4	2.33	97.05	2.27	94.54	2.26	94.16	2.31	96.25
4	3.2	3.13	97.83	3.06	94.63	2.99	93.50	3.09	96.75
5	4.0	3.92	98.01	3.84	96.00	3.76	94.10	3.88	97.08
6	4.8	4.72	95.33	4.64	96.66	4.54	94.58	4.66	97.08

The percentage recoveries along with their fortification level are incorporated in Table-3. The method has advantages over the earlier methods, in that, the dye formation is instantaneous and the recoveries are better, as they are in the range of 95.02-97.80 %. Further, other ingredients of the sample do not interfere and the sensitivity of the method is fairly high.

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