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A Novel Spectrophotometric Method for the Determination of Carbosulfan with 4-Methylaniline

Potla Krishna Murthy, T Niranjan and Nuthalapati Venkatasubba Naidu*

Department of Chemistry, Sri Venkateswara University, Tirupati, Andhra Pradesh, India

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ABSTACT

This study presents Carbosulfan determination by a novel , robustic , mobile, inexpensive, and sensitive spectrophotometric method is developed for the determination of the carbamate pesticide carbosulfan in water and grains . An extractive method, which is based on alkaline hydrolysis of carbosulfan into phenol followed by coupling with diazotized 4- methyl aniline in alkaline medium. Yellowed coloured species (λ_{max} 475 nm) can be extractable in to chloroform, which make reaction more sensitive with same λ_{max} ..Beer's law is obeyed over concentration range of 0.5-10µg/ml. Molar absorptivity and Sandell's sensitivity were found to be 2.292 x 10^4 l $mol^{-1}cm^{-1}$ and 0.0166 µg cm^{-2} respectively. Method has been satisfactory applied for carbosulfan in various environmental samples.

Keywords: carbosulfan, 4-methyl aniline, spectrophotometer.

INTRODUCTION

Carbamate pesticides are essential to agricultural communities; their use has been increased substantially in recent years as a consequence of their selective insecticidal properties and low mammalian toxicity. Hence the number and quantities of carbamate pesticides used in agriculture continue to increase, replacing other type of pesticides such as organochlorine and orgfanophosphorous pesticide [1, 2]. One of the most important carbamate, which is widely used in agriculture, is carbosulfan. In view of its wide application there is a need for the development of sensitive and reliable methods for the quantification of the insecticidal residues in environmental samples.

Carbosulfan, carbamic acid [(dibutyl amino)] 2, 3-dihydro-dimethyl 7-benzofuranyl ester (Shown in Fig. I) is a recently developed broad spectrum systematic and contact insecticide belongs to the class of carbamate insecticide. It undergoes hydrolysis in aqueous medium. Carbosulfan is unstable in acid medium and it will be converted in to carbofuran by the cleavage

of N-S bond, but it is stable under the neutral and alkaline medium [3]. Carbosulfan is used for the control of soil dwelling insects and foliar pests on maize potatoes and sugar beet and used as a nematicide. It is toxic to honeybees, birds and mammals. It acts as a cholinesterase inhibitor. The residue of these insecticide causes air and water pollution. Clay .et.al [4], have reported the activity of the compound.

Literature survey revealed that very few analyzed method such as GC [5, 6], HPLC [7-11] TLC [12-15] and some spectrophotometric methods [16-23] has been reported. The present communication involves the method based on azo-coupling with 4-methylaniline was used as coupling agent which forms yellow colored derivative with the phenol of the insecticide produced by hydrolysis.

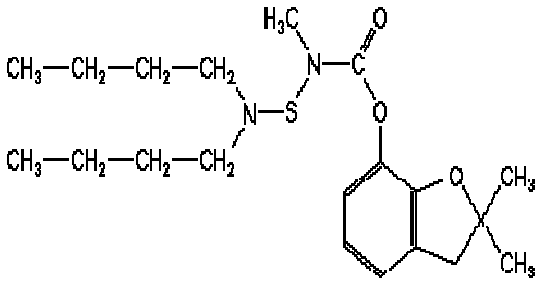

Fig 1: Structure of Carbosulfan

EXPERIMENTAL SECTION

All chemicals used were analytical grade and all of the solutions were freshly prepared with distilled water. A Hitachi, mode U-3400, UV-Vis-NIR spectrophotomer with 1 cm glass cells was used for absorbance measurement.

Sodium hydroxide (2%)
2g of sodium hydroxide was dissolved in 100ml of distilled water.

Sodium nitrite (0.5%)
0.5g of sodium nitrite was dissolved in distilled water and diluted to 100ml.

4-methylaniline (0.2%)
0.2g of 4-methylaniline was dissolved in methanol and diluted to 100ml with distilled water.

Preparation of standard solution of carbosulfan
Stock solution of the above pesticide was prepared by dissolving 50mg of analytical grade insecticide in 100ml of methanol.10 ml of prepared solution was taken into 100 ml standard flask made up to mark to get 50µg/ml.

Preparation of sample solution
Formulation
Carbosulfan 25% wet table powder. An amount equivalent to 50 mg of the insecticide was dissolved in 50 ml of methanol. 5 ml aliquots of the solution were subsequently diluted with 100 ml of methanol.

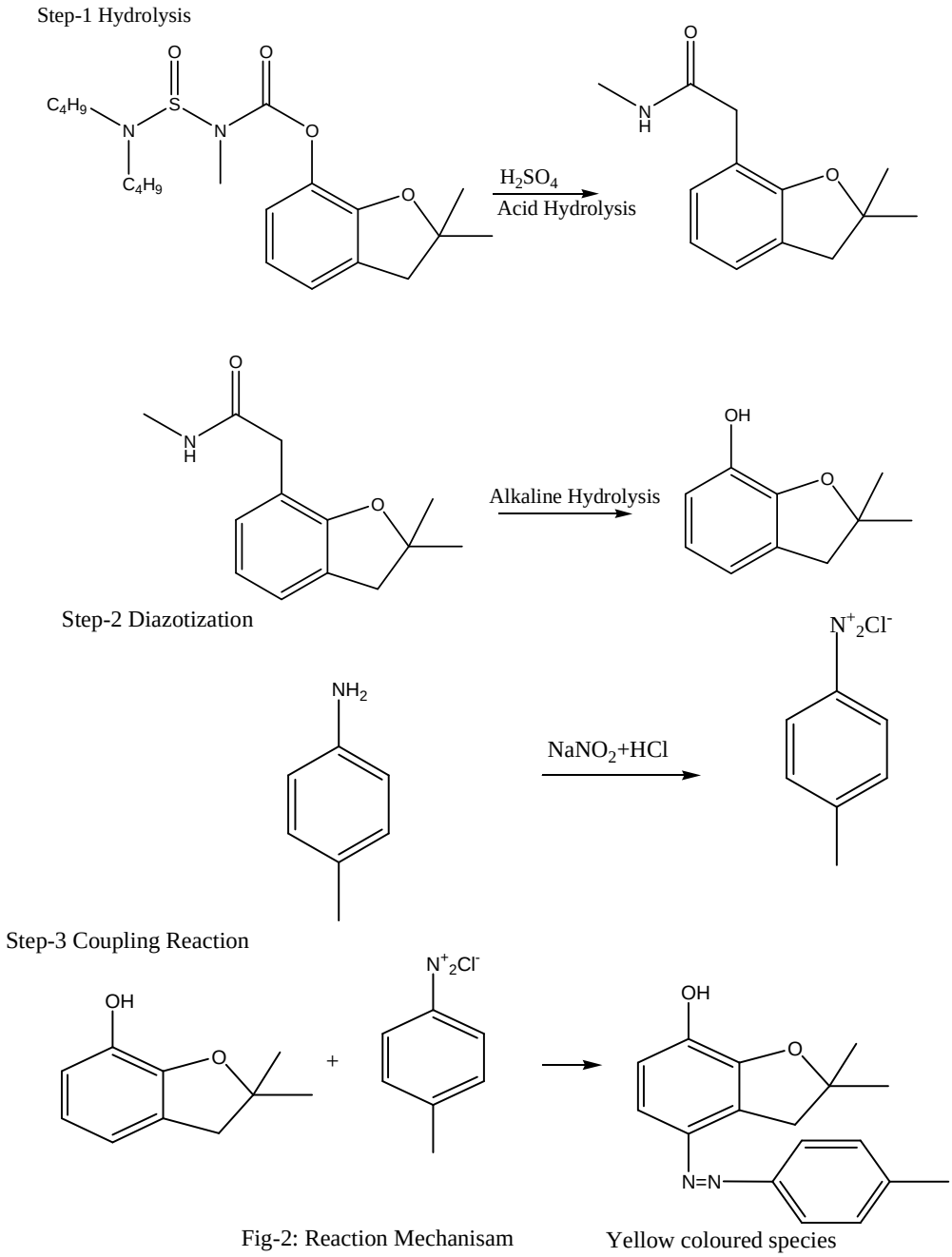

Fig-2: Reaction Mechanisam

General procedure

Aliquots of standard insecticide solution (50µg/ml) 0, 0.25, 0.5, 1.0…….6ml were placed in to a series of 25 ml standard flasks followed by adding 2.5 ml of 2%sodium hydroxide and 2.4 ml diazotization mixture. The solutions were made up to the mark with distilled water and absorbance was measured for the yellow colored chromospheres against a reagent blank. The absorbance is plotted against the final concentration to obtain a calibration graph.

Water samples

pH of each water sample was adjusted to 3-4 with 20% sulfuric acid. One liter sample of distilled water and tap water was fortified with different concentrations of insecticide dissolved in methanol. The fortified water samples were extracted with 100 ml chloroform using a separating funnel. Chloroform extracts were collected in to a funnel and re-extracted the aqueous phase twice with 50 ml chloroform. The combined extracts were washed with 0.1ml potassium

carbonate solution and dried over anhydrous sodium sulphate in a filter funnel and collected the extracts in a 250 ml standard flask. Finally these were made up to the mark with chloroform known aliquots of the chloroform were evaporated to dryness on a steam bath. The residue was dissolved in methanol, and then developed the color.

Grains

100 mg of grains (rice or wheat) was taken in a conical flask and shaken for 5 min with 200 ml chloroform was filtered in to a 520ml standard flask through a what's man no.41 filter paper and residue was washed twice with 10 ml chloroform. Chloroform extracts were combined and made up to the mark known aliquots of chloroform extracts were used for color development after evaporating chloroform.

RESULTS AND DISCUSSION

Optical characteristics, precision and accuracy of the method using diazotized 4-methylaniline as coupling agent

Table 1: Characteristics of the method

Compound	Carbosulfan	
	Present work	**Rajeswari and Naidu[19]**
Concentration range, µg/ml	0.5-10	1-10
Stability of the colored species, hr	72	8
Relative standard deviation, %	0.92	0.8
Relative error, %	0.6	0.6

Table 2: Recovery of Carbosulfan from grains and spiked water samples

Carbosulfan			
Recovery, % *			
Sample	**Added, ppm**	**Present work**	**Rajeswari and Naidu[19]**
Water	1.0	97.0 ± 1.6	96.0 ± 1.6
	3.0	96.5 ± 1.0	96.0 ± 1.2
	5.0	96.0 ± 0.8	95.2 ± 0.8
	7.0	95.5 ± 0.6	94.6 ± 0.7
Rice	1.0	97.0 ± 1.6	96.0 ± 1.6
	3.0	96.0 ± 1.1	96.0 ± 1.2
	5.0	95.8 ± 0.7	94.8 ± 0.8
	7.0	94.7 ± 0.5	93.5 ± 0.6
Wheat	1.0	96.0 ± 1.4	95.0 ± 1.4
	3.0	95.5 ± 0.9	94.1 ± 1.0
	5.0	94.3 ± 0.5	93.2 ± 0.7
	7.0	94.0 ± 0.4	92.8 ± 0.5

** Each value is an average ± standard deviation of five determinations*

The absorbance maximum was 475nm for carbosulfan. Beer's law is obeyed over the range 0.5-10 µg/ml. The color develops instantaneously and remains stable up to 72 hrs. The coupling reaction can be done at room temperature.

The suitability of the proposed method was studied by analysis of ten replicate samples containing 5ppm of carbosulfan. The relative error and relative standard deviation are given in table 1.

Formulation contains carbosulfan were analyzed (10 replicate).For a 25% carbosulfan emulsion the mean deviation was 24.55 ± 0.925.

Recovery experiments were performed with known amounts of the compounds added to different samples of grains and water. For water samples a methanol solution of the insecticide was added. Grains samples were spiked by adding a methanol solution of the insecticide to the dry grain and evaporated.

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