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Spectrophotometric Determination of Bendiocarb in Insecticidal Formulations, Grains and Water

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A simple, rapid and sensitive spectrophotometric method is described for the determination of Bendiocarb. The phenol formed due to alkaline hydrolysis of Bendiocarb is made to couple with diazotized *p*-aminobenzene sulfonamide to give coloured species having an absorption maximum at 448 nm. Beer's law is obeyed over the range 0.5 to 12 ppm.

INTRODUCTION

Bendiocarb or Ficam (1,3-benzodioxol-4-ol, 2,2-dimethyl methyl carbamate) [22781–23–3] is belonging to the class of carbamate insecticides. The insecticidal activity of bendiocarb has been summarised by Lemon[1]. It is a wide spectrum insecticide and it has been commercially developed for the control of insects and other anthropod pests.[2] It is found to be highly toxic to honey bees, birds and dogs.[3] As a result of extensive usage there is a possibility of contamination of environment by this insecticide. There are a few chromatographic[4–10], polarographic[11] and spectrophotometric[12–16] methods are described for the determination of Bendiocarb. In this paper we describe here a simple, rapid and sensitive spectrophotometric method, wherein diazotized *p*-aminobenzene sulfonamide formed as coupling agent. The orange coloured complex has been formed with the phenol of the insecticide generated by alkaline hydrolysis.

EXPERIMENTAL

All chemicals used were of A.R. grade only. Bendiocarb was supplied by Rallis India Ltd., Bangalore. *p*-Aminobenzene sulfonamide (0.1% w/v) was freshly prepared in 10% HCl and prepared aqueous solution of sodium nitrite (0.5% w/v) and sodium hydroxide (2% w/v). Standard solution of bendiocarb (250 μg/ml) was prepared by dissolving 125 mg of insecticide in 500 ml of methanol. Diazotized reagent was prepared as: 20 ml of 0.1% *p*-aminobenzene sulfonamide and 10 ml of 0.5% sodium nitrite were taken into a beaker and mixed well.

UV VIS NIR Spectrophotometer model U 3400, Hitachi, Japan, with 1 cm glass cells, Elico digital pH meter and Keroy single pan electronic balance were used.

Procedure

0.05, 0.1, 0.2, 0.3, . . . , 1.2 mL of aliquots of standard insecticide solutions were taken into 25 ml standard flasks. To each flask 3 mL of sodium hydroxide was added and allowed to stand for 3 min to complete hydrolysis. To each of this 1.5 mL of diazotized *p*-aminobenzene sulfonamide was added and made up to the mark with distilled water. The orange coloured chromophore formed had an absorption maximum at 448 nm against a reagent blank. Calibration plot was constructed.

Preparation of samples

Formulations: 100 Mg of 96% wettable powder was dissolved in 25 mL of methanol and centrifuged for 5 min. Supernatant liquid was transferred into 100 ml standard flask. The residue was washed thrice with 10 mL methanol. The washings were collected and made upto the mark with methanol. Known aliquots of the solution were used for colour development as described earlier and absorbance values were recorded.

Grains: 50 G of grains was placed in a waring blender and blended for 5 min with 100 mL chloroform. The samples were fortified with different concentrations of bendiocarb and blended for 3 min. Chloroform was filtered into a 250 mL calibrated flask through a Whatman No. 1 filter paper. The grains were washed thrice with chloroform and the chloroform extracts were combined and made up to 250 mL. Known aliquots of the chloroform extracts were used for colour development after evaporating chloroform on a steam bath and the amount was determined by the procedure described earlier.

Water: pH of each water sample was adjusted to 4 with 2% sulphuric acid and fortified with different concentrations of bendiocarb in methanol. The spiked water samples were extracted with 100 mL chloroform in a separating funnel by shaking the funnel and reextracted the aqueous phase with 50 mL of chloroform. The combined extracts were washed with 0.1 M potassium carbonate solution and dried by passing through 15–20 g of anhydrous sodium sulphate in a filter funnel and collected the extracts into a 250 mL standard flask. Finally these were made up to the mark with chloroform. Known aliquot of the chloroform was taken and evaporated to dryness on a steam bath and the residue was dissolved in methanol and then the amount was determined by the procedure described earlier.

RESULTS AND DISCUSSION

The absorption maximum was at 448 nm for bendiocarb with *p*-aminobenzene sulfonamide (absorption curve is shown in Fig. 1). Beer's law is obeyed over the range 0.5–12 μg/ml. The colour develops instantaneously and remains stable for 24 h. The

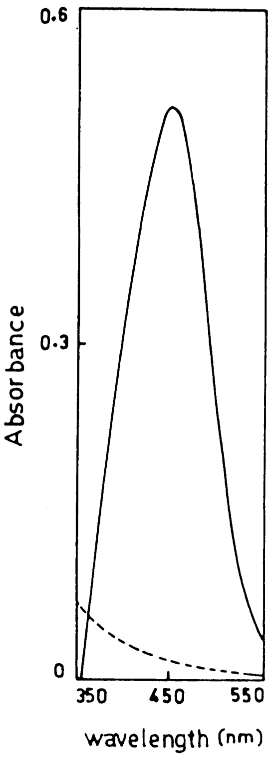

Fig. 1 Bendocarb with *p*-aminobenzene sulphonamide (Absorption spectrum of the complex:______ reagent:- - - - -.

coupling reaction can be done at room temperature. The complex has a molar absorption coefficient of 2.521 × 10 l mol^{-1} cm^{-1}.

The suitability of the proposed method was studied by analysis of six replicate samples containing 5 ppm bendiocarb. The relative standard deviation and Sandell's sensitive values are given in Table 1.

TABLE 1
OPTICAL CHARACTERISTICS OF THE METHOD

1.	Concentration range	0.5–12 μg ml^{-1}
2.	λ_{max}	448 nm
3.	Stability of colour	24 h
4.	Molar absorptivity	2.572 × 10 l mol^{-1} cm^{-1}
5.	Sandell's sensitivity	0.0087 μg cm^{-2}
6.	*Relative standard deviation	0.42
7.	Regression equation	–0.0039 + 0.1143 C
8.	Slope	0.1143
9.	Correlation coefficient	0.9999
10.	Error %	0.35

*Calculated for ten samples containing same amount of bendiocarb.

96% Technical grade sample was analysed. The recoveries are in the range of 95.06 ± 0.34.

Recovery experiments were performed with known amounts of the insecticide added to different samples of water and grains. For water samples a methanol solution of the insecticide was added. Grain samples were spiked by adding a methanol solution of the insecticide to the dry grains and evaporating the solvent.

The results presented in Tables 2 and 3 suggest that the method is applicable for field water samples.

TABLE 2
RECOVERY OF BENDIOCARB ADDED TO GRAINS AND WATER

Sample	Added ppm	Recovery	
		Present work	D.V. Naidu and P.R. Naidu[16]
Rice	1	0.98 ± 0.01	0.97 ± 0.02
	3	2.81 ± 0.04	2.80 ± 0.05
	5	4.82 ± 0.02	4.80 ± 0.04
	7	6.70 ± 0.03	6.70 ± 0.03
Wheat	1	0.97 ± 0.05	0.96 ± 0.03
	3	2.86 ± 0.02	2.85 ± 0.05
	5	4.75 ± 0.04	4.76 ± 0.05
	7	6.66 ± 0.03	6.65 ± 0.07
Water	1	0.97 ± 0.02	0.97 ± 0.02
	3	2.91 ± 0.04	2.90 ± 0.05
	5	4.82 ± 0.03	4.80 ± 0.04
	7	6.72 ± 0.05	6.72 ± 0.06

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