



Trade Science Inc.

April 2008

Volume 7 Issue 6

# Analytical CHEMISTRY

An Indian Journal

Full Paper

ACAIJ 7(6) 2008 [342-345]

## Methodology for estimation of carbosulfan in rice, wheat and water samples

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Received: 12<sup>th</sup> March, 2008 ; Accepted: 17<sup>th</sup> March, 2008

### ABSTRACT

A simple, rapid, sensitive and accurate spectrophotometric method is developed for the determination of carbosulfan in water samples and grains. The method is based on the formation of coloured species upon coupling their hydrolysis product with diazotized 4-aminobenzaldehyde, exhibiting maximum absorption at 484nm. The method obeys Beer's law over the concentration range 0.5-12 $\mu$ g/ml. © 2008 Trade Science Inc. - INDIA

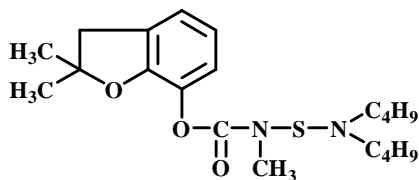
### KEYWORDS

Spectrophotometry;  
Carbosulfan determination;  
4-aminobenzaldehyde  
chloroform.

### INTRODUCTION

Carbosulfan, carbamic acid [(dibutylamino)thio] methyl-2,3 dihydro-2,2 -dimethyl-7-benzofuranyl ester, belongs to the carbamate class of insecticide. It is a recently developed systemic and contact insecticide. It undergoes hydrolysis in aqueous media. Carbosulfan is unstable in acid medium and it will be converted into carbofuran by the cleavage of N-S bond, but is stable under neutral and alkaline conditions<sup>1</sup>. It is used for the control of a wide range of soil-dwelling insects and foliar insect pests on a wide range of crops, like cotton, sugar beet, potato, rice, fruit, citrus, maize, vegetables, sugar cane and coffee. Maitlen and Sladen<sup>2</sup> described the insecticidal activity of carbosulfan. Though it is an insecticide, it has the property of generating the toxicity

among birds and mammals. Clay et al.<sup>3</sup>, described the activity of carbosulfan and metabolic of carbofuran in soils. Umetsu et al.<sup>4</sup>, have reported the activity of carbosulfan in cotton and corn. Raghupathy et al.<sup>5</sup>, described the dissipation of insecticide applied to control plufella xylostella cortis and spodoptera in cauliflower. Creeger<sup>6</sup> described US, EDA procedures for the evaluation of the potential of pesticides to reach ground water. Bruce et al.<sup>7</sup>, described GLC method for the determination of carbosulfan residues along with carbofuran in plants, soil and water. Wilde et al.<sup>8</sup>, have reported the absence of synergism with insecticide combinations used along with carbofurn on chinch bugs. A simple colorimetric method was described by Rajeswari and Naidu<sup>9</sup> for the determination of carbamates in insecticidal formulations and spiked water samples including carbosulfan using diazotised p-aminobenzoic acid as a coupling agent. Naidu and Naidu<sup>10</sup> described a simple spectropotometric technique for the determination of carbosulfan and propoxur with 2-amino benzophenone as coupling agent in water samples and grains. Hamdy soltan<sup>11</sup> described a new colorimetric method for the determination of the carbosulfan resi



dues in distilled, irrigating and drainage underground water using p-aminobenzenesulphonic acid. Reay and Walsh<sup>[12]</sup> reported the damage of *Pinus radiata* seedlings in second rotation forests by the controlled release of carbosulfan. Manjubashini et al.<sup>[13]</sup>, described a spectrophotometric technique for the determination of carbosulfan in various environments. Vassilakis et al.<sup>[14]</sup>, described HPLC technique with post-column fluorescence for the determination of carbosulfan in oranges. Saraswathi et al.<sup>[15]</sup>, developed spectrophotometric method for the determination of carbosulfan and propoxur in environmental samples employing p-amino acetanilide.

The present communication reports a new spectrophotometric method for the determination of the carbosulfan based on the formation of a coloured species by coupling the corresponding phenol obtained by alkaline hydrolysis with diazotized 4-amino benzaldehyde.

## MATERIALS AND METHODS

All chemicals used were of analytical grade and all of the solutions were freshly prepared with distilled water. Elico SL-159 UV VIS Spectrophotometer with 10mm path length glass cells were used for absorbance measurement. Sodium hydroxide (2%) and sodium nitrite (0.5%) solutions were freshly prepared. 4-amino benzaldehyde solution (0.2%) is prepared in 100ml methanol.

### Preparation of standard solution of carbosulfan (50µg/ml)

Stock solution of the above pesticide was prepared by dissolving 50 mg of analytical grade (93.2%) insecticide in 100 ml of methanol. From this working standard solution is prepared by diluting 10 ml of this solution to 100ml.

### Preparation of sample solution

**Formulations:** carbosulfan 25% seed treat. 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.

### Preparation of diazotization mixture

It is prepared by dissolving 18 ml of (0.2%) 4-

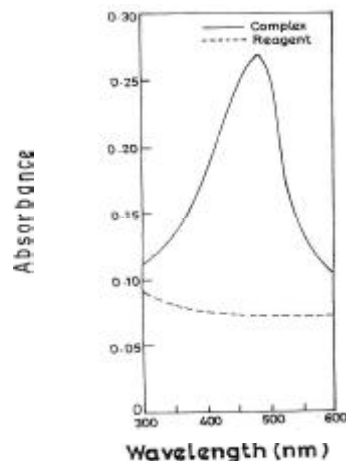


Figure 1: Absorption spectrum of the complex carbosulfan with 4-amino benzaldehyde

aminobenzaldehyde and 30 ml of (0.5%) sodium nitrite solution.

### General procedure

Aliquots of standard insecticide solution 0, 0.25, 0.5, 1.0 ---- 6 ml were placed in series of 25 ml standard flasks followed by adding 2.5 ml of 2% sodium hydroxide and 2.4 ml of diazotized reagent. These solutions were made up to the mark with distilled water and absorbance was measured for the yellow coloured chromophore 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% sulphuric acid. One litre samples of distilled water and tap water was fortified with different concentrations of insecticide dissolved in methanol. The fortified water samples were extracted with 100ml chloroform using a separating funnel. Chloroform extracts were collected into a funnel and are extracted the aqueous phase twice with 50 ml chloroform. The combined extracts were washed with 0.1 M 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 colour.

### Grains

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**TABLE 1: Optical characteristics, precision and accuracy of the method using diazotized 4aminobenzaldehyde as coupling agent**

|                                       |  |
|---------------------------------------|--|
| Beer's law Concentration range        | 1 - 12 $\mu\text{g ml}^{-1}$                 |
| $\lambda$ max                         | 484 nm                                       |
| Stability of the colour               | 48 hrs                                       |
| Molar absorptivity                    | 1.683 $\times 10^4$ l mol $^{-1}$ cm $^{-1}$ |
| Sandell's sensitivity                 | 0.0226 $\mu\text{g cm}^{-2}$                 |
| * Relative standard deviation (RSD %) | 0.51   |
| Regression equation                   | - 0.0322 + 0.0674C                           |
| Slope                                 | 0.0674                                       |
| Correlation coefficient               | 0.9997                                       |
| Error %                               | 1.03   |

\*Calculated for ten samples containing same amount of carbamate

**TABLE 2: Determination of carbosulfan in insecticidal formulations using diazotised 4-aminobenzaldehyde as a coupling agent**

| Labelled amount sample no. | 25% Seed treat | 93.2% Technical grade |
|----------------------------|----------------|-----------------------|
| 1                          | 24.88          | 92.74                 |
| 2                          | 24.84          | 92.76                 |
| 3                          | 24.98          | 92.89                 |
| 4                          | 24.94          | 92.68                 |
| 5                          | 24.97          | 92.78                 |
| 6                          | 24.93          | 92.78                 |
| 7                          | 24.98          | 92.95                 |
| 8                          | 24.96          | 92.93                 |
| Av                         | 24.93          | 92.81                 |
| SD                         | $\pm 0.050$    | $\pm 0.097$           |

**TABLE 3: Recovery of carbosulfan from fortified water samples using diazotized 4-aminobenzaldehyde as a coupling agent**

| Sample no. | Fortification level $\mu\text{g ml}^{-1}$ | Distilled water                        |                      | Tap water                              |                      |
|------------|---|--|----------------------|--|----------------------|
|            |   | Amount recovered $\mu\text{g ml}^{-1}$ | Recovery* percentage | Amount recovered $\mu\text{g ml}^{-1}$ | Recovery* percentage |
| 1          | 1.2                                       | 1.18                                   | 98.3                 | 1.16                                   | 96.6                 |
| 2          | 2.4                                       | 2.34                                   | 97.5                 | 2.34                                   | 97.5                 |
| 3          | 3.6                                       | 3.58                                   | 99.4                 | 3.48                                   | 96.6                 |
| 4          | 4.8                                       | 4.68                                   | 97.5                 | 4.68                                   | 97.5                 |
| 5          | 6.0                                       | 5.87                                   | 97.8                 | 5.71                                   | 95.1                 |
| 6          | 7.2                                       | 7.12                                   | 98.8                 | 6.95                                   | 96.5                 |

\*Mean  $\pm$  S.D. of five determinations

**TABLE 4: Recovery of carbosulfan from fortified grains using diazotized 4-aminobenzaldehyde as a coupling agent**

| Sample no. | Fortification level $\mu\text{g ml}^{-1}$ | Rice                                   |                      | Wheat                                  |                      |
|------------|---|--|----------------------|--|----------------------|
|            |   | Amount recovered $\mu\text{g ml}^{-1}$ | Recovery* percentage | Amount recovered $\mu\text{g ml}^{-1}$ | Recovery* percentage |
| 1          | 0.5                                       | 0.48                                   | 96.0                 | 0.47                                   | 94.0                 |
| 2          | 1.0                                       | 0.95                                   | 95.0                 | 0.96                                   | 96.0                 |
| 3          | 1.5                                       | 1.43                                   | 95.3                 | 1.45                                   | 96.6                 |
| 4          | 2.0                                       | 1.94                                   | 97.0                 | 1.92                                   | 96.0                 |
| 5          | 2.5                                       | 2.45                                   | 96.0                 | 2.41                                   | 96.4                 |
| 6          | 3.0                                       | 2.91                                   | 97.0                 | 2.89                                   | 96.3                 |

\*Mean  $\pm$  S.D. of five determinations

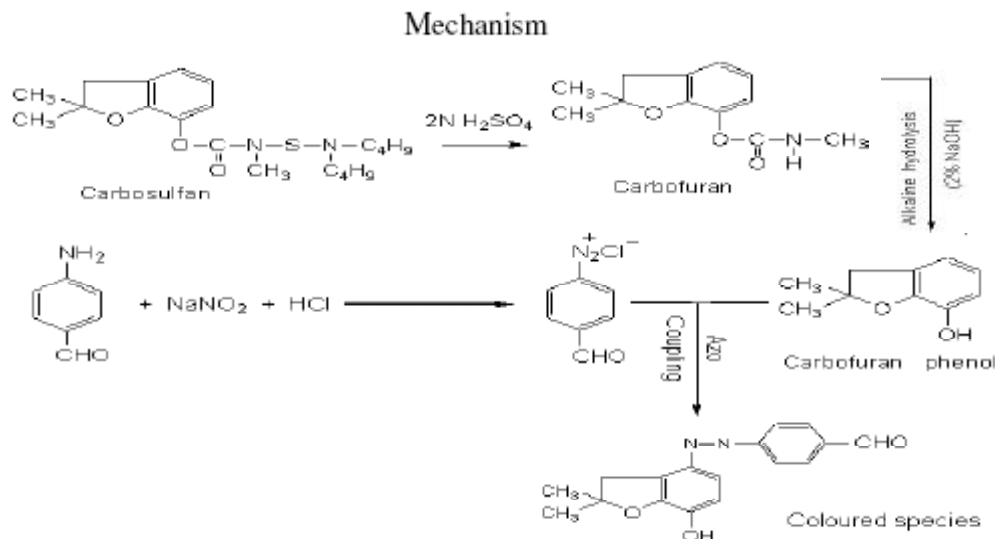
100 g of grains (rice and wheat) was taken in a conical flask and shaken for 5 min. with 200 ml chloroform. Chloroform was filtered into a 250 ml standard flask through a Whatman No.1 filter paper and residue was washed twice with 10 ml chloroform. Chloroform extracts were combined and made up to the mark. Known aliquots of the chloroform extracts were used for colour development after evaporating chloroform.

## RESULTS AND DISCUSSIONS

The absorbance maximum was at 484 nm for carbosulfan. Beer's law is obeyed over the range 1-12 $\mu\text{g/ml}$  for carbosulfan. The colour develops instantaneously and remains stable for more than 48h. The

coupling reaction can be done at room temperature.

The suitability of the proposed method was studied by analysis of ten replicate samples containing 5 ppm of carbosulfan. Recovery experiments were performed with known amounts of the compounds added to different samples of grains and water. The relative error and relative deviations are given in TABLE 1. Formulations containing carbosulfan were analysed (10 replicates) for 25% carbosulfan emulsion the mean  $\pm$  standard deviation was 0.51. The results presented in TABLE 2 show that recovery was in the range 98 to 99%. The results in TABLE 3 and 4 suggest that the method is applicable for the analysis of field water samples also. The method is rapid, sensitive, accurate and cost effective.



### ACKNOWLEDGMENTS

The authors are thankful to M/s FMC- Rallis India Ltd., Bangalore for providing the analytical and technical grade samples of the insecticide

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