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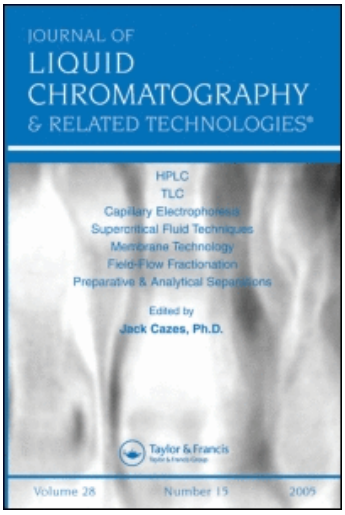

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HPLC Determination of Carbendazim in Formulations

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HPLC DETERMINATION OF CARBENDAZIM IN FORMULATIONS

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ABSTRACT

A rapid high performance liquid chromatographic method has been developed to determine Carbendazim (methyl 2-benzimidazole carbamate) in formulations using reverse phase μ Bondapak C_{18} column and ultraviolet detection. The sample is extracted from formulations with methanol, carbaryl is used as an internal standard. Absorbance is measured at 254 nm and the compound is quantitated by peak height ratios. The method is simple and recoveries averaged between 91-93%.

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INTRODUCTION

Carbendazim (methyl 2-benzimidahole carbamate) is a systematic fungicide controlling a wide range of plant pathogens. Determination of carbendazim has largely been carried out by thin layer chromatography[1,2], gas chromatography[3] and fluorometry[4,5]. Residues of carbendazim in various crops have been studied using liquid chromatography[6,7,8]. Carbendazim has been determined as a degradation product of benomyl (methyl 1-(butylcarbamoyl) - 2-benzimidahole carbamate) by spectrophotometry at low temperatures[9] and HPLC[10,11].

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The analysis of carbamates has been found to be difficult by GC due to severe adsorption and/or thermal degradation[12] while a lack of sensitivity and/or specificity precludes their direct analysis by spectrophotometry[13]. Therefore, HPLC can be considered as a better choice for their analysis.

The present paper describes a simple and rapid method for the direct determination of carbendazim in formulations by reverse phase HPLC using UV detector. To the best of authors knowledge, such studies have not been reported earlier.

EXPERIMENTAL

Liquid chromatograph and UV detector (Model 48) were from Waters Associates Ltd. The column used was μ Bondapak C_{18}/Porasil B, 3.9 mm x 30 cm i.d., stainless steel. Filtration assembly was from Millipore Corp., Bedford, MA. All the solvents were of HPLC grade and were obtained from Spectrochem Pvt. Ltd., Bombay.

A. Carbendazim (25 mg), technical grade, was dissolved in methanol (100 ml).

B. Carbaryl (250 mg), technical grade, was dissolved in methanol (500 ml). This solution was used as the internal standard.

C. Carbendazim (25 mg), technical grade, was dissolved in solution B (100 ml).

D. Commercial formulations, weighed so as to contain Ca 25 mg Carbendazim, were dissolved in solution B (100 ml) by agitating on a magnetic stirrer for 1 hr. The solution was filtered through Millipore Assembly and the filtrate was used for HPLC analysis.

3 replicate injections of each of the solutions A, B, C and D were made. Wavelength mobile-phase and flow-rates were varied so as to optimize

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conditions for best resolution of the chromatographic peaks.

Operating conditions were: Flow-rate 1 ml min^{-1}; wavelength 254 nm; mobile phase acetonitrile-water 60:40; detector range 0.1 AUFS; injection volume 10 µl.

RESULTS AND DISCUSSION

A flow-rate of 1 ml min^{-1}, keeping the wavelength fixed at 254 nm, was found to be the best for satisfactory resolution. The two peaks (Carbendazim and Carbaryl) were observed to be well resolved when a 60:40 mixture of acetonitrile-water was used as the mobile phase. This mixture gave low UV background interference.

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Peak heights of standards and samples were measured, Pesticide content in formulations was determined as follows:

$$\%\text{Carbendazim} = (R/R^1) \times (W^1/W) \times P$$

where,

R & R^1 = average peak height ratios for samples and standards respectively.

W^1 = mg Carbendazim in std. soln.

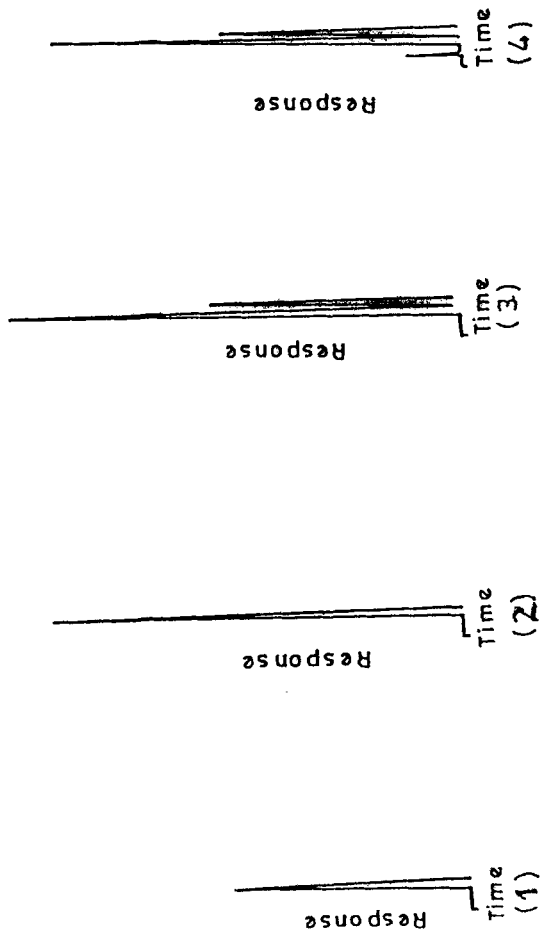

Fig. 1 : Liquid chromatograms of carbendazim on reverse-phase Bondapak C_{18} column, mobilephase 60 :40 acetonitrite-water. 1, Soln B; 2, Soln A; 3, Soln C; 4, Soln D;

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W = mg Carbendazim (Ca 25 mg) in sample solution.

P = % purity of standard.

The recoveries in both the formulations ranged between 91-93% (Fig.1).

The formulation recovery was checked by mixing equal volumes of solutions C and D, (containing 250 mg l^{-1} of carbendazim) and measuring their peak heights. Further, equal volumes of carbendazim (125 mg l^{-1}) and a formulation (containing Ca 125 mg l^{-1} carbendazim) in solution B, were mixed and their peak heights were measured. It was found that the peak heights for carbendazim were exactly half in the latter case, while carbaryl peak retained its original height.

The method reported is very sensitive and can be successfully applied for the identification and quantitation of carbendazim in samples obtained from different sources, in very low concentrations.

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