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Thin-layer chromatographic behaviour of carbamate pesticides and related compounds

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ABSTRACT

The thin-layer chromatographic behaviour of carbaryl, carbendazim (Bavistin), carbofuran, propoxur, phenol, 4-chlorophenol, *o*-nitrophenol, α -naphthol and β -naphthol was examined on alumina, barium sulphate, calcium carbonate, calcium phosphate, calcium sulphate, cellulose and silica gel G in solvents such as acetone, benzene, carbon tetrachloride, chloroform, ethanol and distilled water. The important separations achieved were carbendazim from carbaryl, propoxur, 4-chlorophenol, *o*-nitrophenol, α -naphthol and β -naphthol; carbaryl, propoxur, α -naphthol and β -naphthol from carbofuran, phenol, 4-chlorophenol and *o*-nitrophenol; and *o*-nitrophenol from carbofuran, phenol and 4-chlorophenol.

INTRODUCTION

Thin-layer chromatography (TLC) is used for the qualitative and quantitative analysis of a wide variety of compounds [1]. Pandalikar *et al.* [2] have developed a plain thin-layer chromatographic (P-TLC) procedure for the detection of carbaryl at trace levels in biological fluids. Srivastava and Recna [3] developed a suitable P-TLC scheme for the separation of carbaryl, bendiocarb, carbofuran, baygon, ziram, zineb, aldicarb, 2-isopropyl-phenyl-*N*-methylcarbamate (MIPC) and 2-sec.-butyl-*N*-methylcarbamate (BPMC) on plates coated with silica gel containing 1% zinc acetate using benzene-ethyl acetate (50:10) as the solvent. Residues of carbofuran and its two carbamate metabolites have been extracted with HCl, partitioned into CH₂Cl₂, chromatographed on silica gel and detected with KOH-*p*-nitrobenzenediazonium fluoroborate [4].

Our previous work showed that papers impregnated with an ion-pair reagent or reversed-phase reagent can be used for the separation of herbicides and plant growth regulators [5]. P-TLC and sequen-

tial TLC (S-TLC) can be used for the detection and determination of carbaryl in water [6,7]. In continuation of our previous work, the chromatographic behaviour of carbamate pesticides and related compounds has been examined on thin layers of alumina, barium sulphate, calcium carbonate, calcium phosphate, calcium sulphate, cellulose and silica gel G.

EXPERIMENTAL

Apparatus

A Stahl apparatus with a universal applicator (thickness of the applied layers adjustable from 0.25 to 2.00 mm), glass plates (20 × 4 cm), glass jars (25 × 5 cm), a hot-air drier, a temperature-controlled electric oven (Tempo), a Spectronic-20 spectrophotometer (Bausch and Lomb), an electrical hot-plate with magnetic stirrer (Sunvic) and a micropipette (10–100 μ l) (Gilson) were used.

Reagents and chemicals

Compounds were of laboratory-reagent (LR), general-reagent (GR), wettable powder (WP) or analytical-reagent (AR) grade as indicated. Aluminium hydroxide gel (Qualigens), barium sulphate (Ranbaxy), calcium carbonate and calcium phos-

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phate (LR) (GSC), calcium sulphate dihydrate (Merck), microcrystalline cellulose (LR) (CDH), silica gel G (Merck), 50% carbaryl (WP) (Paushak), 3% carbofuran (GR) (Pesticides), 1% (w/w) propoxur (Bayer), 50% Bavistin (WP) (BASF), phenol, *p*-chlorophenol (LR) (BDH), *o*-nitrophenol (LR) (CDH), α - and β -naphthol (AR) (CDH), 4-nitrobenzenediazonium tetrafluoroborate (Merck), potassium hydroxide (LR) (CDH), sulphanilic acid, sodium nitrite (AR) (CDH) and sodium hydroxide (LR) (Qualigens) were used. All other reagents were of analytical-reagent grade.

Preparation of solutions

Solutions (1%) of carbaryl, carbendazim, carbofuran, propoxur, phenol, 4-chlorophenol, *o*-nitrophenol, α -naphthol and β -naphthol were prepared in ethanol and 4-nitrobenzenediazonium tetrafluoroborate solution (0.1%) was prepared in acetone. Methanolic potassium hydroxide (1%) and aqueous solutions of sodium nitrite (0.3%) and sodium hydroxide (16%) were used. Sulphanilic acid (0.2%) solution was prepared in 10% HCl. When a 1% solution of a pesticide could not be prepared, the saturated solution was used.

Preparation of TLC plates

The following slurries were applied to the glass plates with the help of the applicator so that the thickness of the coating was 0.5 mm: (1) aluminium hydroxide gel (30 g) in distilled water (DW) (100 ml); (2) barium sulphate (30 g) in DW (60 ml); (3) calcium carbonate (25 g) in DW (60 ml); (4) calcium phosphate (40 g) in DW (60 ml); (5) calcium sulphate (30 g) in DW (70 ml); (6) microcrystalline cellulose (20 g) in DW (75 ml); and (7) silica gel G (25 g) in DW (65 ml).

The plates were first allowed to dry at room temperature ($25 \pm 2^\circ\text{C}$) and then in an oven at 110°C for 30 min for the cellulose coating and 1 h for the other coatings for activation.

Spotting of test solution

Test solutions were applied to the TLC plates with a fine capillary or micropipette. After drying at room temperature, the plates were developed to a distance of 10 cm with a suitable mobile phase. The solvents investigated, with dielectric constants [8] in parentheses, were hexane (1.89), 1,4-dioxane (2.21),

carbon tetrachloride (2.24), benzene (2.28), chloroform (4.81), chlorobenzene (5.71), ethyl acetate (6.02), acetone (20.7), ethanol (24.3), methanol (32.63), nitrobenzene (34.82) and distilled water (78.54).

Detection of pesticides

The carbamates and phenols were detected on the TLC plates by spraying first with potassium hydroxide solution and then with *p*-nitrobenzenediazonium tetrafluoroborate solution. Coloured spots on a white background appeared for all the compounds.

Recording of R_F values

For tailing spots, the distance from the origin for the leading (R_L) and trailing (R_T) fronts were measured and R_m values were calculated with the equation

$$R_m = \frac{\text{average of } R_L \text{ and } R_T \text{ (cm)}}{\text{distance travelled by solvent front (cm)}}$$

For compact spots, R_F values were calculated in the conventional manner [5], *i.e.*, R_F = migration distance relative to solvent front.

Qualitative separations

To achieve the separation of pesticides, one of the compounds was spotted, the solvent was removed, the second compound was spotted, the solvent was removed again, then the plate was developed, dried and pesticides were located as above.

Quantitative separations

The pesticide solutions were spotted on fresh plates, solvents were removed and the plates were developed as above. The previously demarcated area of the plate was scratched off and carbaryl was extracted with methanol (5 ml) and determined by the following procedure [9].

Spectrophotometric determination of carbaryl

To the carbaryl extract (5 ml) in a 50-ml volumetric flask, 10 ml of distilled water and 2 ml each of sodium nitrite and sulphanilic acid solutions were added. After 10 min, 5 ml of 16% NaOH were added and the solution was made up to the mark with distilled water and mixed thoroughly. The absorbance was recorded against a blank after 10 min at 520 nm. The blank contained all the reagents except carbaryl.

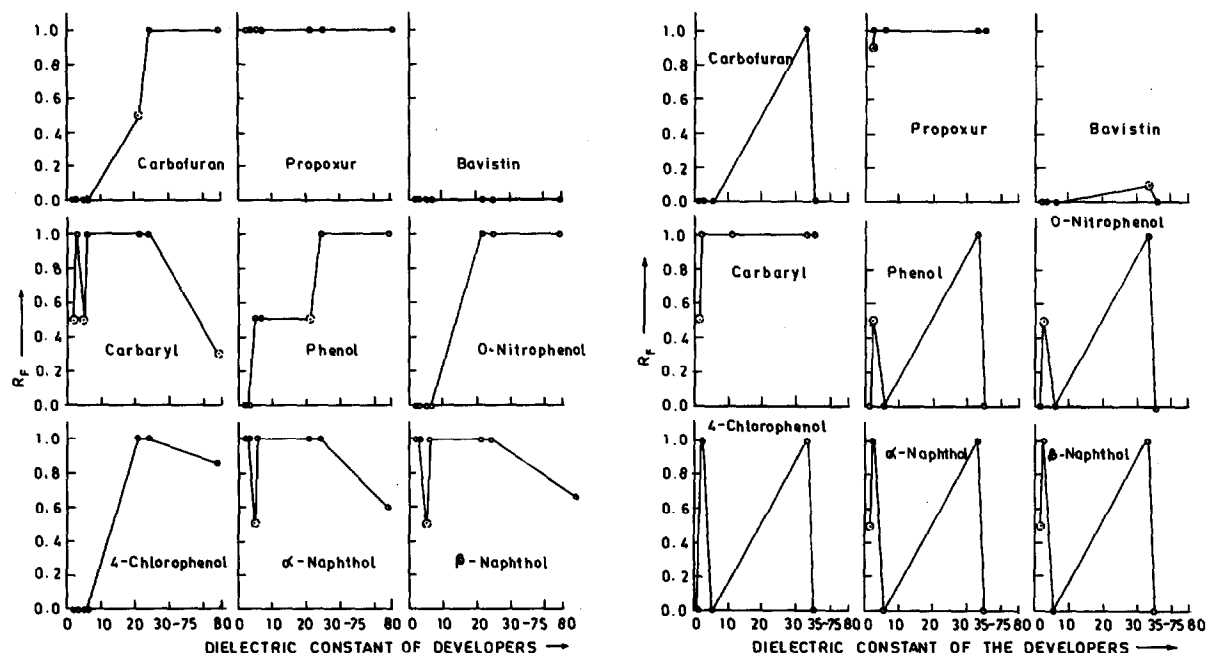


Fig. 1. TLC behaviour of some carbamates and related compounds on cellulose using different solvents. Solid circles, R_F ; dashed circles, R_m .

RESULTS

Plots of R_F values versus the dielectric constants of the solvents are shown in Figs. 1-7. The separations

achieved are recorded in Tables I-VII. Thin-layer chromatograms of some analytically important separations are shown in Fig. 8. Analytical results for the quantitative separations are summarized in

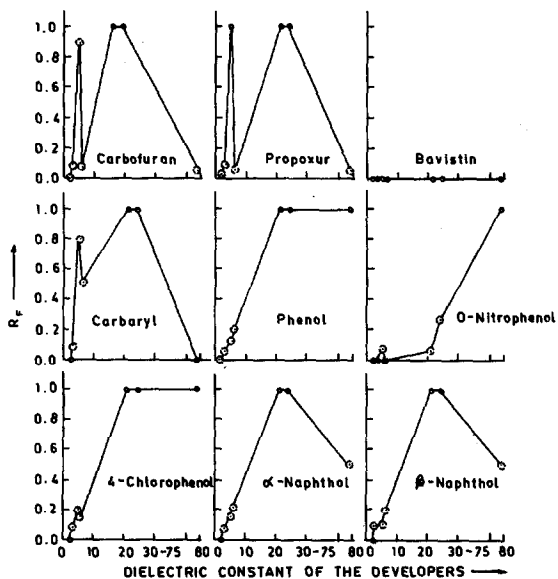


Fig. 2. TLC behaviour of some carbamates and related compounds on aluminium hydroxide gel. Symbols as in Fig. 1.

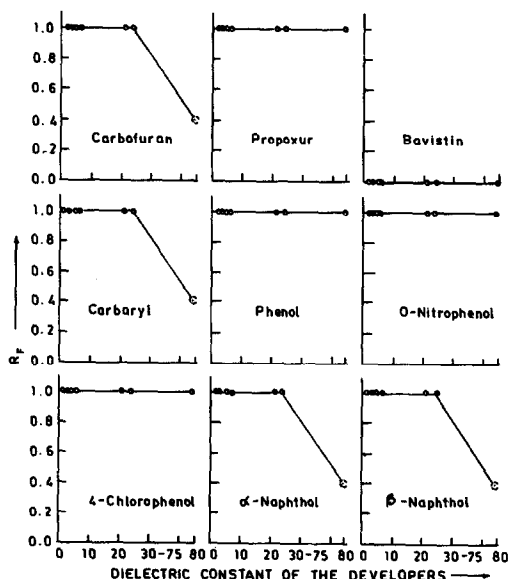


Fig. 3. TLC behaviour of some carbamates and related compounds on barium sulphate. Symbols as in Fig. 1.

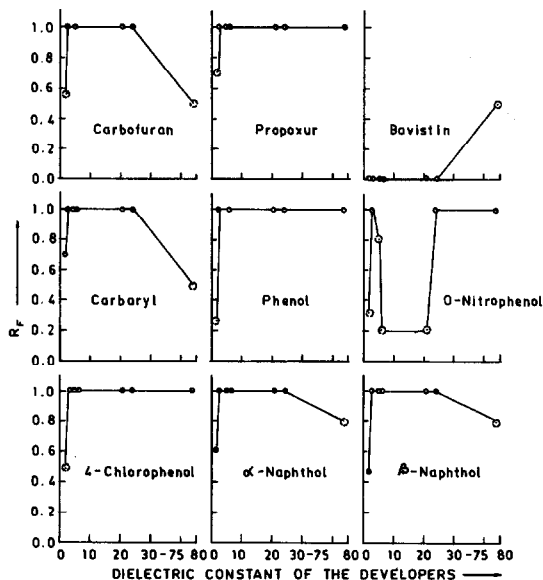


Fig. 4. TLC behaviour of some carbamates and related compounds on calcium carbonate. Symbols as in Fig. 1.

Table VIII. The times required for developing the chromatograms with different coatings are given in Table IX.

Statistical analysis of the spectrophotometric data was applied using the following expressions:

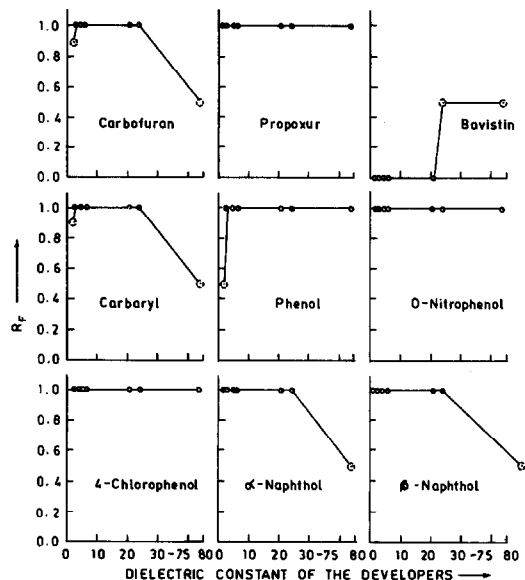


Fig. 6. TLC behaviour of some carbamates and related compounds on calcium sulphate. Symbols as in Fig. 1.

$$\sigma = \sqrt{\frac{(x_1 - \mu)^2 + (x_2 - \mu)^2 + (x_3 - \mu)^2 + \dots}{N - 1}}$$

$$\text{R.S.D.} = \frac{\sigma \cdot 100}{\mu}$$

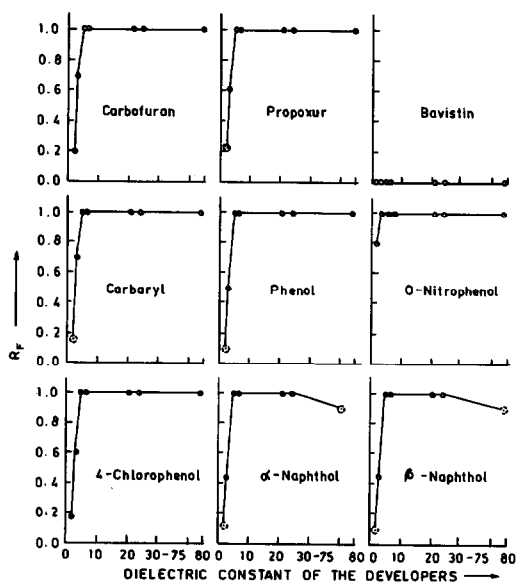


Fig. 5. TLC behaviour of some carbamates and related compounds on calcium phosphate. Symbols as in Fig. 1.

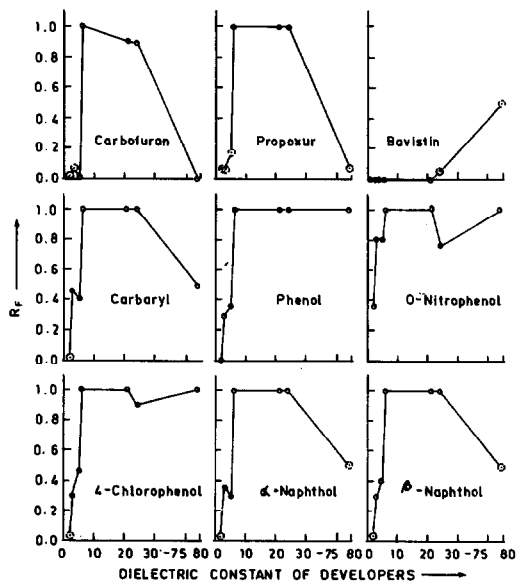


Fig. 7. TLC behaviour of some carbamates and related compounds on silica gel G. Symbols as in Fig. 1.

TABLE I
SEPARATIONS ACHIEVED IN DIFFERENT SOLVENTS ON ALUMINIUM HYDROXIDE GEL

Compounds ^a	Separated from ^a	Solvent
Bavistin (0.0) and <i>o</i> -nitrophenol (0-1 cm)	Carbaryl (1.0), carbofuran (1.0), 4-chlorophenol (1.0), α -naphthol (1.0), β -naphthol (1.0), phenol (1.0) and propoxur (0.9)	Acetone
Carbaryl (7-9 cm), carbofuran (1.0) and propoxur (1.0)	Bavistin (0.0), 4-chlorophenol (0-3 cm), <i>o</i> -nitrophenol (0-1.5 cm) and phenol (0-2.5 cm)	Chloroform
4-Chlorophenol (1.0), <i>o</i> -nitrophenol (1.0) and phenol (1.0)	Bavistin (0.0), carbaryl (0.0), carbofuran (0-1 cm) and propoxur (0-2 cm)	Distilled water
Bavistin (0.0) and <i>o</i> -nitrophenol (1-4 cm)	Carbaryl (1.0), carbofuran (1.0), 4-chlorophenol (1.0), α -naphthol (1.0), β -naphthol (1.0), phenol (1.0) and propoxur (1.0)	Ethanol

^a R_F values or migration distances (cm) are given in parentheses.

TABLE II
SEPARATIONS ACHIEVED IN DIFFERENT SOLVENTS ON BARIUM SULPHATE

Compound ^a	Separated from ^a	Solvent
Bavistin (0.0)	Carbaryl (1.0), carbofuran (1.0), 4-chlorophenol (1.0), α -naphthol (1.0), β -naphthol (1.0), <i>o</i> -nitrophenol (1.0), phenol (1.0) and propoxur (1.0)	Acetone
Bavistin (0.0)	Carbaryl (1.0), carbofuran (1.0), 4-chlorophenol (1.0), α -naphthol (1.0), β -naphthol (1.0), <i>o</i> -nitrophenol (1.0), phenol (1.0) and propoxur (1.0)	Benzene
Bavistin (0.0)	Carbaryl (1.0), carbofuran (1.0), 4-chlorophenol (1.0), α -naphthol (1.0), β -naphthol (1.0), <i>o</i> -nitrophenol (1.0), phenol (1.0) and propoxur (1.0)	Carbon tetrachloride
Bavistin (0.0)	Carbaryl (1.0), carbofuran (1.0), 4-chlorophenol (1.0), α -naphthol (1.0), β -naphthol (1.0), <i>o</i> -nitrophenol (1.0), phenol (1.0) and propoxur (1.0)	Chloroform
Bavistin (0.0)	4-Chlorophenol (1.0), <i>o</i> -nitrophenol (1.0), phenol (1.0) and propoxur (1.0)	Distilled water
Bavistin (0.0)	Carbaryl (1.0), carbofuran (1.0), 4-chlorophenol (1.0), α -naphthol (1.0), β -naphthol (1.0), <i>o</i> -nitrophenol (1.0), phenol (1.0) and propoxur (1.0)	Ethanol
Bavistin (0.0)	Carbaryl (1.0), carbofuran (1.0), 4-chlorophenol (1.0), α -naphthol (1.0), β -naphthol (1.0), <i>o</i> -nitrophenol (1.0), phenol (1.0) and propoxur (1.0)	Ethyl acetate

^a R_F values or migration distances (cm) are given in parentheses.

TABLE III
SEPARATIONS ACHIEVED IN DIFFERENT SOLVENTS ON CALCIUM CARBONATE

Compounds ^a	Separated from ^a	Solvent
Bavistin (0.0) and <i>o</i> -nitrophenol (0-4 cm)	Carbaryl (1.0), carbofuran (1.0), 4-chlorophenol (1.0), α -naphthol (1.0), β -naphthol (1.0), phenol (1.0) and propoxur (1.0)	Acetone
Bavistin (0.0)	Carbaryl (1.0), carbofuran (1.0), 4-chlorophenol (1.0), α -naphthol (1.0), β -naphthol (1.0), <i>o</i> -nitrophenol (1.0), phenol (1.0) and propoxur (1.0)	Benzene
Bavistin (0.0)	Carbaryl (1.0), carbofuran (4-7 cm), 4-chlorophenol (2-7 cm), α -naphthol (0.6), β -naphthol (0.45) and propoxur (0.7)	Carbon tetrachloride
Bavistin (0.0)	Carbaryl (1.0), carbofuran (1.0), 4-chlorophenol (1.0), α -naphthol (1.0), β -naphthol (1.0), phenol (1.0) and propoxur (1.0)	Chloroform
Bavistin (0.0)	Carbaryl (1.0), carbofuran (1.0), 4-chlorophenol (1.0), α -naphthol (1.0), β -naphthol (1.0), phenol (1.0) and propoxur (1.0)	Ethanol
Bavistin (0.0) and <i>o</i> -nitrophenol (0-4 cm)	Carbaryl (1.0), carbofuran (1.0), 4-chlorophenol (1.0), α -naphthol (1.0), β -naphthol (1.0), phenol (1.0) and propoxur (1.0)	Ethyl acetate

^a R_F values or migration distances (cm) are given in parentheses.

TABLE IV
SEPARATIONS ACHIEVED IN DIFFERENT SOLVENTS ON CALCIUM PHOSPHATE

Compound ^a	Separated from ^a	Solvent
Bavistin (0.0)	Carbaryl (1.0), carbofuran (1.0), 4-chlorophenol (1.0), α -naphthol (1.0), β -naphthol (1.0), <i>o</i> -nitrophenol (1.0), phenol (1.0) and propoxur (1.0)	Acetone
Bavistin (0.0)	Carbaryl (1.0), carbofuran (1.0), 4-chlorophenol (1.0), α -naphthol (0.7), β -naphthol (0.45), <i>o</i> -nitrophenol (1.0), phenol (0.5) and propoxur (0.6)	Benzene
<i>o</i> -Nitrophenol (0.8)	Bavistin (0.0), carbaryl (0-2 cm), carbofuran (0.2), 4-chlorophenol (0.15), α -naphthol (0-2.5 cm), β -naphthol (0-2 cm), phenol (0-2 cm) and propoxur (0.25)	Carbon tetrachloride
Bavistin (0.0)	Carbaryl (1.0), carbofuran (1.0), 4-chlorophenol (1.0), α -naphthol (1.0), β -naphthol (1.0), <i>o</i> -nitrophenol (1.0), phenol (1.0) and propoxur (1.0)	Chloroform
Bavistin (0.0)	Carbaryl (1.0), carbofuran (1.0), 4-chlorophenol (1.0), α -naphthol (1.0), β -naphthol (1.0), <i>o</i> -nitrophenol (1.0), phenol (1.0) and propoxur (1.0)	Distilled water
Bavistin (0.0)	Carbaryl (1.0), carbofuran (1.0), 4-chlorophenol (1.0), α -naphthol (1.0), β -naphthol (1.0), <i>o</i> -nitrophenol (1.0), phenol (1.0) and propoxur (1.0)	Ethanol
Bavistin (0.0)	Carbaryl (1.0), carbofuran (1.0), 4-chlorophenol (1.0), α -naphthol (1.0), β -naphthol (1.0), <i>o</i> -nitrophenol (1.0), phenol (1.0) and propoxur (1.0)	Ethyl acetate

^a R_F values or migration distances (cm) are given in parentheses.

where x_1, x_2, x_3 , etc. = measured values, μ = average value, N = number of sets, σ = standard deviation and R.S.D. = relative coefficient of variation standard deviation.

DISCUSSION

Fig. 1-7 show that amongst the seven adsorbents studied aluminium hydroxide gel possesses a critical

separation potential, *i.e.*, the pattern of R_F values is entirely different to those on barium sulphate, calcium carbonate, calcium phosphate and calcium sulphate. The TLC behaviours of silica gel G, cellulose and aluminium hydroxide gel are very similar; the degree of polarity of the adsorbents may be responsible for this deviation.

Tables I-VII show that several separations can be achieved on these adsorbents. The definite size and

TABLE V
SEPARATIONS ACHIEVED IN DIFFERENT SOLVENTS ON CALCIUM SULPHATE

Compound ^a	Separated from ^a	Solvent
Bavistin (0.0)	Carbaryl (1.0), carbofuran (1.0), 4-chlorophenol (1.0), α -naphthol (1.0), β -naphthol (1.0), <i>o</i> -nitrophenol (1.0), phenol (1.0) and propoxur (1.0)	Acetone
Bavistin (0.0)	Carbaryl (1.0), carbofuran (1.0), 4-chlorophenol (1.0), α -naphthol (1.0), β -naphthol (1.0), <i>o</i> -nitrophenol (1.0), phenol (1.0) and propoxur (1.0)	Benzene
Bavistin (0.0)	Carbaryl (1.0), carbofuran (1.0), 4-chlorophenol (1.0), α -naphthol (1.0), β -naphthol (1.0), <i>o</i> -nitrophenol (1.0), phenol (1.0) and propoxur (1.0)	Carbon tetrachloride
Bavistin (0.0)	Carbaryl (1.0), carbofuran (1.0), 4-chlorophenol (1.0), α -naphthol (1.0), β -naphthol (1.0), <i>o</i> -nitrophenol (1.0), phenol (1.0) and propoxur (1.0)	Chloroform
Bavistin (0.0)	Carbaryl (1.0), carbofuran (1.0), 4-chlorophenol (1.0), α -naphthol (1.0), β -naphthol (1.0), <i>o</i> -nitrophenol (1.0), phenol (1.0) and propoxur (1.0)	Ethyl acetate

^a R_F values or migration distances (cm) are given in parentheses.

TABLE VI
SEPARATIONS ACHIEVED IN DIFFERENT SOLVENTS ON CELLULOSE

Compounds ^a	Separated from ^a	Solvent
Bavistin (0.0)	Carbaryl (1.0), chlorophenol (1.0), α -naphthol (1.0), β -naphthol (1.0), <i>o</i> -nitrophenol (1.0) and propoxur (1.0)	Acetone
Carbaryl (1.0), α -naphthol (1.0), β -naphthol (1.0) and propoxur (1.0)	Bavistin (0.0), carbofuran (0.0), 4-chlorophenol (0.0), <i>o</i> -nitrophenol (0.0) and phenol (0.0)	Benzene
α -Naphthol (1.0), β -naphthol (1.0) and propoxur (1.0)	Bavistin (0.0), carbofuran (0.0), 4-chlorophenol (0.0), <i>o</i> -nitrophenol (0.0) and phenol (0.0)	Carbon tetrachloride
Carbaryl (1.0) and propoxur (1.0)	Bavistin (0.0), carbofuran (0.0), 4-chlorophenol (0.0), α -naphthol (0.0), β -naphthol (0.0), <i>o</i> -nitrophenol (0.0) and phenol (0.0)	Chlorobenzene
Propoxur (1.0)	Bavistin (0.0), carbofuran (0.0), 4-chlorophenol (0.0) and <i>o</i> -nitrophenol (0.0)	Chloroform
Bavistin (0.0) and carbofuran (0.0)	Carbaryl (1.0), 4-chlorophenol (1.0), α -naphthol (1.0), β -naphthol (1.0) and propoxur (1.0)	Dioxan
Bavistin (0.0)	Carbaryl (1.0), carbofuran (1.0), 4-chlorophenol (1.0), α -naphthol (1.0), β -naphthol (1.0), <i>o</i> -nitrophenol (1.0), phenol (1.0) and propoxur (1.0)	Ethanol
Bavistin (0-2 cm), carbaryl (0-6 cm), α -naphthol (0.6) and β -naphthol (0.55)	Carbofuran (1.0), <i>o</i> -nitrophenol (1.0), phenol (1.0) and propoxur (1.0)	Distilled water
Bavistin (0-2 cm)	α -Naphthol (0.6) and β -naphthol (0.55)	Distilled water
Carbaryl (1.0), α -naphthol (1.0), β -naphthol (1.0) and propoxur (1.0)	Bavistin (0.0), carbofuran (0.0), 4-chlorophenol (0.0) and <i>o</i> -nitrophenol (0.0)	Ethyl acetate
Propoxur (1.0)	Bavistin (0.0), carbofuran (0.0), 4-chlorophenol (0.0), <i>o</i> -nitrophenol (0.0) and phenol (0.0)	Hexane
Bavistin (0-2 cm)	Carbaryl (1.0), carbofuran (1.0), 4-chlorophenol (1.0), α -naphthol (1.0), β -naphthol (1.0), <i>o</i> -nitrophenol (1.0), phenol (1.0) and propoxur (1.0)	Methanol
Carbaryl (1.0) and propoxur (1.0)	Bavistin (0.0), carbofuran (0.0), 4-chlorophenol (0.0), α -naphthol (0.0), β -naphthol (0.0), <i>o</i> -nitrophenol (0.0) and phenol (0.0)	Nitrobenzene
Bavistin (0-1 cm), carbaryl (0-6 cm), α -naphthol (0.6) and β -naphthol (0.5)	Carbofuran (1.0), 4-chlorophenol (1.0), <i>o</i> -nitrophenol (1.0), phenol (1.0) and propoxur (1.0)	Tap water
Bavistin (0-1 cm)	α -Naphthol (0.6) and β -naphthol (0.5)	Tap water

^a R_F values or migration distances (cm) are given in parentheses.

TABLE VII
SEPARATIONS ACHIEVED IN DIFFERENT SOLVENTS ON SILICA GEL G

Compounds ^a	Separated from ^a	Solvent
Bavistin (0.0)	Carbaryl (1.0), carbofuran (0.9), 4-chlorophenol (1.0), α -naphthol (1.0), β -naphthol (1.0) <i>o</i> -nitrophenol (1.0), phenol (1.0) and propoxur (1.0)	Acetone
<i>o</i> -Nitrophenol (0.80)	Bavistin (0.0), carbaryl (0.45), carbofuran (0.0), 4-chlorophenol (0.30), α -naphthol (0.35), β -naphthol (0.30), phenol (0.30) and propoxur (0-1 cm)	Benzene
<i>o</i> -Nitrophenol (0.40)	Bavistin (0.0), carbaryl (0-0.05 cm), carbofuran (0.05), 4-chlorophenol (0-0.05 cm), phenol (0.0) and propoxur (0-1 cm)	Carbon tetrachloride
<i>o</i> -Nitrophenol (0.80)	Bavistin (0.0), carbaryl (0.4), carbofuran (0.6), 4-chlorophenol (0.45), α -naphthol (0.45), β -naphthol (0.50), phenol (0.35) and propoxur (1-0.5 cm)	Chloroform
Carbofuran (0.0) and propoxur (0-1.5 cm)	4-Chlorophenol (1.0), <i>o</i> -nitrophenol (1.0) and phenol (1.0)	Distilled water
Bavistin (0-1 cm)	Carbaryl (1.0), carbofuran (0.9), 4-chlorophenol (0.9), α -naphthol (1.0), β -naphthol (1.0) <i>o</i> -nitrophenol (0.75), phenol (1.0) and propoxur (1.0)	Ethanol
Bavistin (0.0)	Carbaryl (1.0), carbofuran (1.0), 4-chlorophenol (1.0), α -naphthol (1.0), β -naphthol (1.0) <i>o</i> -nitrophenol (1.0), phenol (1.0) and propoxur (1.0)	Ethyl acetate

^a R_F values or migration distances (cm) are given in parentheses.

TABLE VIII

ANALYTICAL PARAMETERS FOR QUANTITATIVE SEPARATIONS OF CARBARYL FROM OTHER PESTICIDES ON CELLULOSE

Amount of carbaryl applied (μg)	Separated from ^a	Carbaryl found (μg)	Developer	Error (%) ^b	$\mu \pm \sigma^b$	R.S.D. (%) ^b
25	Bavistin	22.666	Ethanol	9.36	0.136 ± 0.0058	4.207
50		44.642		10.72	0.250 ± 0.010	4.000
75		70.178		6.43	0.393 ± 0.0057	1.450
100		93.928		6.07	1.254 ± 0.0066	1.254
25	Carbofuran	22.166	Benzene	11.336	0.133 ± 0.011	8.164
50		46.428		7.144	0.26 ± 0.01	3.846
75		70.178		6.429	0.393 ± 0.0208	5.297
100		93.928		6.072	0.526 ± 0.0231	4.393
25	<i>o</i> -Nitrophenol	23.75	Benzene	5.000	0.133 ± 0.00578	4.345
50		45.714		8.572	0.256 ± 0.0058	2.265
75		68.928		8.096	0.386 ± 0.0058	1.502
100		93.392		6.608	0.523 ± 0.0057	1.089
25	Propoxur	22.166	Distilled water	11.336	0.133 ± 0.0057	4.285
50		47.499		5.002	0.266 ± 0.0058	2.180
75		70.178		6.429	0.393 ± 0.0115	2.798
100		93.928		6.072	0.526 ± 0.0152	2.908

^a 5 μg in each instance.^b $N = 4$.

shape of the spots obtained on the TLC plates are shown in Fig. 8. The analytical data in Table VIII show that cellulose is a good TLC material for the clean-up and separation of carbaryl from other materials. TLC detection on cellulose was found to

be much more sensitive than that on the other adsorbents. Table IX shows that the development is fast on cellulose (10 min) but very slow on barium sulphate (1020 min) in carbon tetrachloride. The development times with the different adsorbents

TABLE IX

TIME REQUIRED FOR ASCENDING CHROMATOGRAM (10 cm) IN DIFFERENT SOLVENTS

Coating	Time (min)						
	Acetone	Benzene	Carbon tetrachloride	Chloroform	Distilled water	Ethanol	Ethyl acetate
Aluminium hydroxide gel	25	30	135	95	30	90	30
Barium sulphate	360	510	1020	615	460	1045	375
Calcium carbonate	25	40	65	50	35	70	30
Calcium phosphate	25	35	60	40	50	100	35
Calcium sulphate	10	15	30	18	10	30	15
Cellulose	7	10	15	10	15	25	5
Silica gel G	37	50	125	70	45	145	55

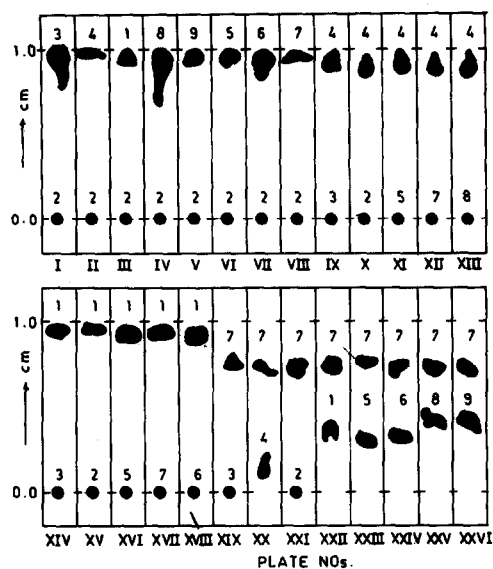


Fig. 8. Separations of some carbamate pesticides and related compounds on cellulose (I–XVIII) and silica gel G (XIX–XXVI) with the developers ethanol (I–VIII), chlorobenzene (IX–XVIII) and chloroform (XIX–XXVI). 1 = Carbaryl; 2 = carbendazim; 3 = carbofuran; 4 = propoxur; 5 = phenol; 6 = 4-chlorophenol; 7 = o-nitrophenol; 8 = α-naphthol; 9 = β-naphthol.

increase in the order cellulose < calcium sulphate < calcium phosphate < calcium carbonate < silica gel G < aluminium hydroxide gel < barium sulphate and the sequence of development with different

solvents is acetone < ethyl acetate < distilled water < benzene < chloroform < carbon tetrachloride < ethanol.

Barium sulphate, calcium sulphate, calcium phosphate and cellulose have a good analytical potential as TLC materials for carbamate pesticides.

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