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LABORATORY PREPARATION AND APPLICATIONS OF MODIFIED CARBOWAX 20M BONDED SUPPORTS TO THE GAS CHROMATOGRAPHY OF PESTICIDES

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SUMMARY

Five gas chromatographic (GC) solid supports, modified by treatment to give surface bonded Carbowax 20M, were evaluated for their suitability by GC stationary phases for several pesticides both before and after coating with OV-210. Even though all the modified supports were far superior to non-treated supports, there were appreciable differences between the supports for some of the more difficult to chromatograph pesticides. Carbowax 20M bonded to Gas-Chrom P generally gave the best performance for the pesticides tested. Preparation of the support for optimum response required extensive refluxing in 9 *N* hydrochloric acid to remove all traces of acid, coating with 5% Carbowax 20M, conditioning overnight at 280° followed by Soxhlet extraction with methyl alcohol and dichloromethane to remove all unbound Carbowax 20M. It was demonstrated that certain modified packings could play a useful role in the analysis of such compounds.

INTRODUCTION

The novel development of polymer-deactivated adsorbents for use in gas–liquid chromatography has been described previously by Hastings and Aue[1]. These authors applied an ultrathin film of Carbowax 20M on gas chromatographic (GC) supports equivalent to a monomolecular layer[2] as they nominally described it. The result of this treatment was a "deactivated" support which greatly improved the GC behavior of polar and/or relatively unstable organic compounds. These authors[1] went one step further by coating the "deactivated" support with different liquid phases. The purpose of these different liquid phases was to improve separations beyond that attainable with Carbowax treatment alone, thereby permitting an analyst to choose selectively that phase(s) which would give desired separations and at the same time be less concerned with the poor chromatographic behavior attributed to non-deactivated supports.

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In the analysis of pesticides, one is faced with many compounds that are very difficult to separate and/or are often unstable on commerically prepared supports. The objective of this study was to determine what factors are most critical in the preparation of Carbowax 20M surface-modified solid supports and the practical application of these supports to pesticide separations.

EXPERIMENTAL

Preparation of supports for gas chromatography

The principle used for preparing the supports was the same as that described by Aue *et al.*[2]. The supports evaluated in this study (Table I) included Chromosorb P, 80–100 mesh (Analabs, North Haven, Conn., U.S.A.); Chromosorb G, 80–100 mesh (Analabs.); acid-washed Chromosorb W, 80–100 mesh (Analabs); Gas-Chrom Q, 80–100 mesh (Applied Science Labs., State College, Pa., U.S.A.); and Gas-Chrom P, 80–100 mesh (Applied Science Labs.). Each commercial diatomaceous earth support was acid washed in a Soxhlet apparatus with 9 *N* hydrochloric acid until all traces of a yellow hue were removed. This generally required a minimum of 1–3 weeks, depending on the support. The supports were then washed to neutrality with distilled water and dried. Each support was gently screened through a 120-mesh Tyler stainless-steel screen to remove fines, and coated with Carbowax 20M (usually 5%) using rotary evaporation until a good uniform coating was achieved.

TABLE I

SUPPORT MATERIALS AS RECEIVED FROM THE MANUFACTURER PRIOR TO MODIFICATION BY CARBOWAX 20M TREATMENT

Source, Diatomite; mesh size, 80–100.

Support	*Color*	*Surface area (m^2/g)*	*Treatment by manufacturer*	*Free fall density (g/ml)*
Chromosorb P	Pink	4.0	Calcined	0.38
Chromosorb G	Oyster white	0.5	–	0.47
Chromosorb W	White	1.0	Flux calcined, acid washed	0.18
Gas-Chrom P	White	1.0	Flux-calcined, acid-washed, base-washed	0.22
Gas-Chrom Q	White	1.0	Flux calcined, acid washed, base washed, DMCS treated	0.22

The coating of the support with the Carbowax was accomplished by mixing the support with Carbowax 20M dissolved in dichloromethane and allowing to stand overnight before evaporating the solvent on a rotary evaporator. Solvent was then added to the coated support, the mixture stirred gently and allowed to set one hour before solvent removal by rotary evaporation. This procedure was carried out two more times before the coating was considered uniform. The dried supports were transferred to a 50-ml volumetric pipette containing a glass wool plug at the restricted end and capped with an additional glass wool plug at the top of the packing. The pipette containing the support was placed in a 4-in. long circular heating block which had an I.D. sufficient to hold the pipette at a flow-rate approximating 200 ml/min

for at least 30 min. The flow of nitrogen was then reduced to 5 ml/min. The temperature was then raised to 270°–280° (unless specified otherwise) over a period of about 2 h and maintained at that temperature for 20 h before cooling to room temperature. The supports were then transferred to a Soxhlet apparatus and successively extracted with methanol and dichloromethane (unless otherwise stated) for at least seven days. All supports were then dried and divided into two portions, one of which was coated with OV-210 and the other used without further treatment for general comparisons. OV-210 coated supports were prepared in the same manner as the Carbowax 20M treated supports.

Pesticides evaluated

The pesticides selected for evaluation (Table II) vary greatly in polarity and suitability as solutes for GC on conventional silicone coated columns[3]. Many of these compounds are generally considered thermally unstable, yield unfavorable separations, and/or give less than optimum chromatographic behavior as often characterized by peak tailing or broadening.

TABLE II

PESTICIDES USED IN THE EVALUATION OF MODIFIED SUPPORTS

Common name	*Chemical name*
Aldrin	1,2,3,4,10,10-Hexachloro-1,4,4*a*,5,8,8*a*-hexahydro-endo-1,4-exo-5,8-dimethanonaphthalene
Atrazine	2-chloro-4-(ethylamino)-6-(isopropylamino)-*s*-triazine
Azinphos-methyl	O,O-Dimethyl S-[4-oxo-1,2,3-benzotriazin-3(4H)-ylmethyl] phosphorodithioate
Benefin	N-Butyl-N-ethyl-α,α,α-trifluoro-2,6-dinitro-*p*-toluidine
Chlorpyrifos	O,O-Diethyl O-(3,5,6-trichloro-2-pyridyl) phosphorothioate
Chlorpyrifos oxygen analogue	O,O-Diethyl O-(3,5,6-trichloro-2-pyridyl) phosphate
Diazinon	O,O-Diethyl O-(2-isopropyl-6-methyl-4-pyrimidinyl) phosphorothioate
Dioxathion	2,3-*p*-Dioxanedithiol-S,S-bis(O,O-diethyl phosphorodithioate)
Disulfoton	O,O-Diethyl-S-2-(ethylthio)-ethyl phosphorodithioate
Lindane	γ-Isomer of 1,2,3,4,5,6-hexachlorocyclohexane
Methamidophos	O,S-Dimethyl phosphoramidothioate
Methyl Parathion	O,O-Dimethyl O-*p*-nitrophenyl phosphorothioate
Mevinphos	O,O-Dimethyl 1-carbomethoxy-1-propen-2-yl phosphate (60% *trans*, 30% *cis* isomer)
Monocrotophos	*cis*-3-(dimethoxyphosphinyloxy)-N-methylcrotonamide
Parathion	O,O-Diethyl-O-*p*-nitrophenyl phosphorothioate
Paraoxon	O,O-Diethyl O-*p*-nitrophenyl phosphate
Phosphamidon	2-Chloro-N,N-diethyl-3-hydroxycrotonamide dimethyl phosphate
p,p'-DDT	1,1,1-Trichloro-2,2-bis(*p*-chlorophenyl) ethane
Simazine	6-Chloro-N,N'-diethyl-1,3,5-triazine-2,4-diamine
Tepp	Tetraethyl pyrophosphate
Trifluralin	α,α,α-Trifluoro-2,6-dinitro-N,N-dipropyl-*p*-toluidine

Gas chromatography

All supports were packed in 1.8 m × 2 mm I.D. U-shaped glass columns and evaluated in a Varian Aerograph Model 2100 gas chromatograph equipped with ^{3}H electron-capture detectors (ECDs) and a Microtek Model 220 gas chromatograph

equipped with a ^{63}N ECD. The Varian Aerograph gas chromatograph was equipped for on-column injection while the Microtek Model 220 was equipped for off-column injection utilizing a demisting trap as received from the manufacturer. Only two column temperatures were used in this study, 175° and 200°, to keep operating conditions as static as possible. Nitrogen was used as the carrier gas and flow-rates were maintained at 25 ml/min. As a precautionary procedure it is very important that the carrier gas be free of oxygen in order to prevent removal of the film through oxidation[4].

RESULTS AND DISCUSSION

The unique behavior of a thin film of Carbowax 20M on a support such as Chromosorb W or G, very adequately described by previous investigators[1,2,5,6], has shown potential for the analysis of a limited number of pesticides[4,5,7]. The primary purpose of this study was to ascertain if a micro-thin layer of Carbowax 20M was useful as a stationary phase for the GC analysis of multi-pesticide residues, and to ascertain more carefully what precautions might be necessary in preparing coated supports for studies involving pesticide residues. Five commonly employed GC supports were selected (Table I) for deactivation using the Carbowax 20M treatment. These treated supports were then compared with non-deactivated supports by observing differences in their ability to efficiently chromatograph unstable and/or difficult to separate pesticides. Gas chromatographs equipped with ^{3}H and ^{63}Ni ECDs were employed in this study because of their wide use and sensitivity to many types of pesticides and their inherent ability to detect degradation products, chromatographic peak tailing, and artifacts not always observed with other GC detection systems. Therefore, it is not unreasonable to believe that if the various columns performed well with the ECD system, they should perform even better with element-selective detectors.

Many of the steps in the original procedure[1,2] for preparing the supports were subjected to systematic variation in this study; examples include the percent load of Carbowax 20M, the choice of solvent used to remove the excess Carbowax, the temperature selected for overnight conditioning of the coated Carbowax on the solid support, and the necessity for the acid treatment. Only one variable was evaluated at a time while the others were kept constant according to the original procedure, unless stated otherwise.

When 2.5 and 10% (w/w) coatings of Carbowax 20M on Chromosorb G were compared, no significant GC differences could be observed. Therefore, this variable was not considered critical and a 5% Carbowax treatment was used for subsequent evaluations.

The next variable tested was the extracting solvent used to remove excess Carbowax 20M. The original extracting solvent was methanol[2]. Since Carbowax 20M is not readily soluble in methanol, dichloromethane was also used. Acid-washed Chromosorb G was coated with 10% Carbowax and heat treated as usual. The material was then divided into two portions. One portion was extracted with methanol and the other with dichloromethane. No differences in chromatographic behavior were observed for the compounds tested except for azinphos-methyl and diazinon. The latter showed a much greater relative retention time (*RRT*) with the methanol-

extracted support but peak shape was improved slightly with the dichloromethane extracted support. Azinphos-methyl appeared to decompose on the dichloromethane treated column. Therefore, a third treatment was carried out by extracting first with methanol for 3 or 4 days followed by dichloromethane for the remainder of a total period of seven days. The dual-solvent treatment gave identical retention times as for the single methanol extraction but at the same time provided the better peak shapes characteristic of the single dichloromethane extraction. Consequently, the dual-solvent extraction was utilized for the remainder of this study.

The temperature used for conditioning the Carbowax coated solid support appeared to be somewhat critical based on previous studies. Aue *et al.*[4] reported that temperatures in excess of 280° resulted in polymer deterioration but gave improved chromatographic behavior for the alcohols and hydrocarbons tested. Two different batches of Carbowax 20M coated on acid-washed Chromosorb G were subjected to 250° and 270° heat treatments. No real differences were observed for the pesticides tested, with the exception that the support from the lower temperature treatment produced an extra peak equivalent to *p,p'*-DDE following injection of DDT. This was not evident with the 270° treated support. Whether this was due to the unique difference in the Carbowax polymer resulting from the heat treatment or a difference in actual distribution of the Carbowax on the diatomaceous earth support is difficult to conclude since no load determinations were attempted.

A fourth aspect of this study challenged the necessity of the initial acid treatment of the solid support prior to Carbowax coating. Since acid-washed supports are commercially available it would be advantageous to omit this process. To evaluate adequately the need for the acid treatment, a commercially prepared batch of Gas-Chrom P (which is acid-washed by the manufacturer) coated with 15% Carbowax 20M was heat treated and solvent extracted. Another batch of Gas-Chrom P was treated similarly except that it was acid washed in this laboratory prior to treatment with Carbowax 20M. For many of the compounds tested the commercially coated packing compared favorably with our acid-washed and coated support (Table III). However, phosphimadon, paraoxon, and azinphos-methyl were not eluted, and mevinphos and *p,p'*-DDT gave broader peaks at different retention times on the commercial phase than with the other packings (Table IV). Several other compounds also gave different *RRT* values on these two supports.

It was important to compare the differences between a commonly employed commercial-prepared packing with the same packing which had been reconditioned prior to coating with the stationary phase. Therefore, a commercial packing of 10% OV-210 on Gas-Chrom Q was compared to a batch of Gas-Chrom Q reconditioned in our laboratory. The Gas-Chrom Q was extracted for four days with methanol–hydrochloric acid (1:1) followed by our usual extraction with hydrochloric acid and Carbowax 20M treatment. The reconditioned Gas-Chrom Q was coated with 5% OV-210 and compared with the commercially prepared OV-210 on Gas-Chrom Q. The commercial packing produced much broader peaks, severe tailing, and different *RRT* values (Table III) for many of the pesticides tested. In general, the laboratory-prepared Carbowax 20M treated packing was far superior. It was quite evident from this portion of the study that the acid treatment of supports greatly improved the chromatographic performance, particularly for many of the difficult to chromatograph pesticides.

TABLE III

THE EFFECT OF VARIOUS TREATMENTS ON THE CHROMATOGRAPHIC BEHAVIOR OF MODIFIED CARBOWAX 20M SUPPORTS

Peak shape is defined by numbers: 1 = sharp peak with little or no tailing; 2 = sharp but tailing; 3 = broad but symmetrical with little or no tailing; 4 = moderate tailing; 5 = severe tailing; N.P. = no peak. *RRT* = relative retention time (parathion = 1.00). Gas-Chrom P columns were operated at 175°; Gas-Chrom Q columns were operated at 200°.

Pesticide	*Commercial Gas-Chrom P coated with 15% Carbowax 20M, heat treated and solvent extracted*		*Acid-washed Gas-Chrom P coated with Carbowax 20M, heat treated and solvent extracted*		*Commercial Gas-Chrom Q coated with 10% OV-210*		*Modified Gas-Chrom Q acid washed, heat treated, solvent extracted, and coated with 5% OV-210*	
	Peak shape	*RRT*	*Peak shape*	*RRT*	*Peak shape*	*RRT*	*Peak shape*	*RRT*
Phosphamidon	N.P.	—	1	0.52, 0.78	2	0.10	4	0.12, 0.15
Mevinphos	3	0.13	1	0.14	5	0.20	1	0.17
Methamidophos	3	0.23	3	0.28	—	—	—	—
Diazinon	1	0.23	1	0.29	1	0.17	1	0.15
Lindane	1	0.31	1	0.31	1	0.17	1	0.26
Disulfoton	1	0.23	1	0.29	1	0.21, 0.72	1	0.27
Atrazine	1	0.63	1	0.65	2	0.20	1	0.30
Simazine	1	0.98	1	0.82	2	0.20	1	0.32
Benefin	1	0.10	1	0.17	—	—	—	—
Trifluralin	1	0.10	1	0.17	—	—	—	—
Aldrin	1	0.24	1	0.26	1	0.21	1	0.29
Dioxathion	3	0.52	3	0.37	2	0.23, 0.44	1	0.32
Chlorpyrifos	1	0.55	1	0.55	1	0.38	1	0.46
Monocrotophos	4	0.34, 0.43	4	1.49	—	—	—	—
Methyl Parathion	1	1.01	1	0.90	4	0.74	1	0.80
Parathion	1	1.00	1	1.00	1	1.00	1	1.00
Chlorpyrifos oxygen analogue	1	1.90	1	0.78	—	—	—	—
p,p'-DDT	3	1 01	1	2.39	—	—	—	—
Paraoxon	N.P.	—	1	1.04	5	1.69	1	1.33
TEPP	1	2.56	1	2.51	—	—	—	—
Azinphos-methyl	N.P.	—	1	13.00	4	4.55	3	7.10

TABLE IV

PEAK SHAPE AND RELATIVE RETENTION ON UNCOATED CARBOWAX 20M-MODIFIED SUPPORTS

Peak shape is defined by numbers: 1 = sharp peak with little or no tailing; 2 = sharp but tailing; 3 = broad but symmetrical with little or no tailing; 4 = moderate tailing; 5 = severe tailing; 6 = peak poorly distinguished; N.P. = no peak. *RRT* = relative retention time (Parathion = 1.00). All column temperatures were held constant at 175° with the exception of Chromosorb P which had a column temperature of 220°.

Pesticide	*Chromosorb P*		*Chromosorb G*		*Chromosorb W*		*Gas-Chrom P*		*Gas-Chrom Q*	
	Peak shape	*RRT*	*Peak shape*	*RRT*	*Peak shape*	*RRT*	*Peak shape*	*RRT*	*Peak shape*	*RRT*
Phosphamidon	4	0.13	*	0.05, 0.07	1	0.08	1	0.52, 0.78	1	0.11, 0.85
Mevinphos	1	0.21	1	0.12	1	0.16	1	0.14	1	0.14
Methamidophos	4	0.17	4	0.32	4	0.62	3	0.28	4	0.18
Diazinon	3	0.24	3	0.31	1	0.37	1	0.29	1	0.35
Lindane	1	0.27	3	0.26	1	0.24	1	0.31	1	0.31
Disulfoton	1	0.28	3	0.28	1	0.31	1	0.29	1	0.30
Atrazine	4	0.32	3	0.61	4	0.62	1	0.65	1	0.65
Simazine	4	0.33	3	0.72	4	0.70	1	0.82	1	0.86
Benefin	1	0.29	1	0.15	1	0.18	1	0.17	1	0.18
Trifluralin	1	0.29	1	0.14	1	0.18	1	0.17	1	0.18
Aldrin	1	0.32	3	0.25	1	0.26	1	0.26	1	0.28
Dioxathion	1	0.32	3	0.35	4	0.36	3	0.37	1	0.38
Chlorpyrifos	1	0.46	3	0.59	1	0.60	1	0.55	1	0.56
Monocrotophos	4	0.78	4	0.16, 1.48	5	2.22	2	1.49	2	1.46
Methyl Parathion	1	0.82	3	0.84	1	0.72	1	0.90	1	0.90
Parathion	1	1.00	3	1.00	1	1.00	1	1.00	1	1.00
Chlorpyrifos oxygen analogue	6	—	6	—	6	—	1	0.78	1	0.72, 1.84
p,p'-DDT	3	1.11	3	2.32	3	0.82, 1.99	1	2.39		2.34
Paraoxon	N.P.	—	3	1.06	4	1.35	1	1.04	1	1.06
TEPP	1	2.36	3	2.90	1	3.17	1	2.51	1	2.58
Azinphos-methyl	N.P.	—	3	12.06	3	10.17	1	13.00	1	11.75

* Peaks on a solvent front; could not distinguish peak shape.

TABLE V

PEAK SHAPE AND RELATIVE RETENTION ON CARBOWAX 20M MODIFIED SUPPORTS COATED WITH OV-210

Peak shape is defined by numbers: 1 = sharp peak with little or no tailing; 3 = broad but symmetrical with little or no tailing; 4 = moderate tailing; 5 = severe tailing; N.P. = no peak. *RRT* = relative retention time (Parathion = 1.00). All column temperatures were held constant at 200°.

Pesticide	*10% OV-210 on Chromosorb P*		*10% OV-210 on Chromosorb G*		*5% OV-210 on Chromosorb W*		*5% OV-210 on Gas-Chrom P*		*5% OV-210 on Gas-Chrom Q*	
	Peak shape	*RRT*	*Peak shape*	*RRT*	*Peak shape*	*RRT*	*Peak shape*	*RRT*	*Peak shape*	*RRT*
Phosphamidon	3	0.10	1	0.10, 0.12	1	0.08, 0.10	1	0.12, 0.14	4	0.12, 0.15
Mevinphos	3	0.15	1	0.15	1	0.17	1	0.17	1	0.17
Methamidophos	—	—	3	0.15	5	0.17	3	0.15	4	0.15, 0.26
Diazinon	3	0.20	1	0.23	1	0.20	1	0.21	1	0.15
Lindane	3	0.23	1	0.24	1	0.23	1	0.25	1	0.26
Disulfoton	3	0.24	1	0.25	1	0.25	1	0.26	1	0.27
Atrazine	3	0.29	1	0.30	1	0.26	1	0.28	1	0.30
Simazine	3	0.33	1	0.34	1	0.28	1	0.29	1	0.32
Benefin	—	—	1	0.25	1	0.28	1	0.29	1	0.28
Trifluralin	—	—	1	0.25	1	0.29	1	0.29	1	0.28
Aldrin	3	0.27	1	0.28	1	0.28	1	0.29	1	0.29
Dioxathion	3	0.28	3	0.30	1	0.29	1	0.31	1	0.32
Chlorpyrifos	3	0.43	1	0.45	1	0.44	1	0.46	1	0.46
Monocrotophos	—	—	3	0.25, 0.84	4	0.76	3	0.71	4	0.26, 0.74
Methyl Parathion	3	0.79	1	0.80	1	0.78	1	0.80	1	0.80
Parathion	3	1.00	1	1.00	1	1.00	1	1.00	1	1.00
Chlorpyrifos oxygen analogue	—	—	3	0.86	*		1	0.23, 1.00	*	
p,p'-DDT	3	1.18	3	1.26	1	1.06	1	1.15	1	0.62, 1.17
Paraoxon	N.P.	N.P.	1	1.30	1	1.35	1	1.34	1	1.33
TEPP	—	—	3	1.47	1	1.40	1	1.41	1	1.39
Azinphos-methyl	—	—	1	7.36	1	5.74	1	6.06	3	7.10

* Very poor response with many small peaks and a large hump.

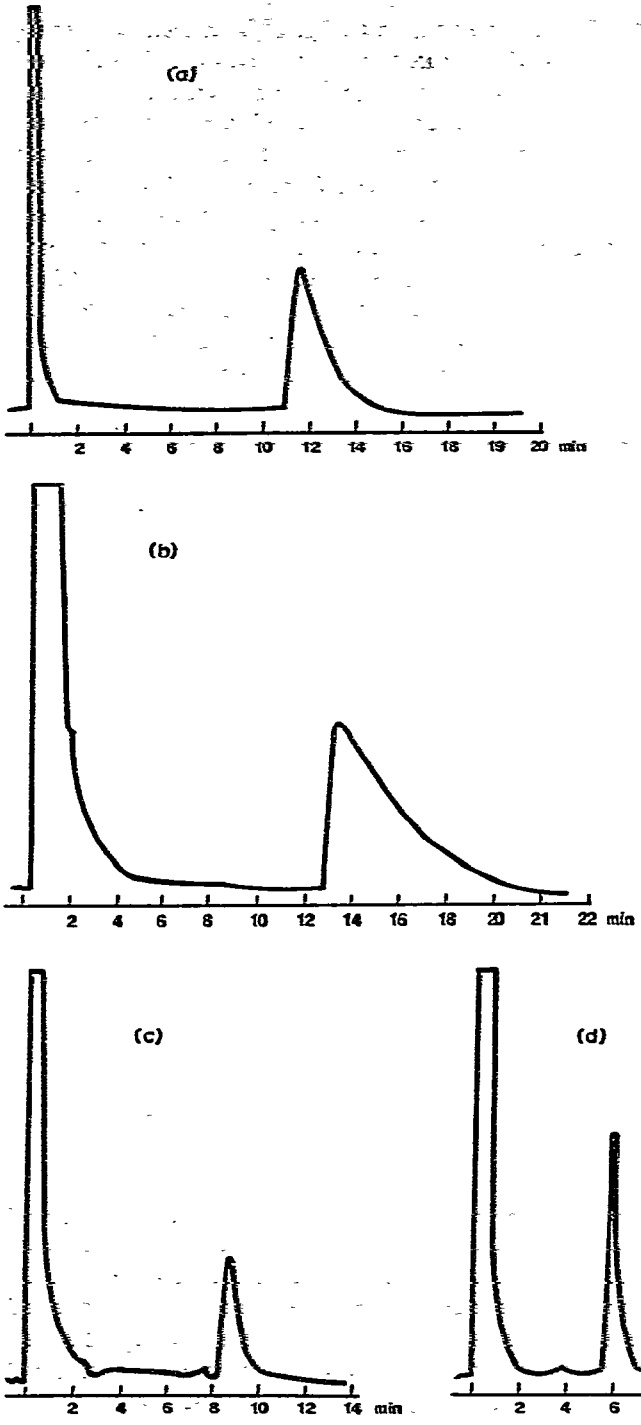

Fig. 1. Chromatogram of monocrotophos on Carbowax 20M-modified (a) Chromosorb G, (b) Chromosorb W, (c) Gas-Chrom Q, and (d) Gas-Chrom P. Column temperature, 175°.

Tables IV and V compare *RRT* values and chromatographic efficiencies for 21 pesticides on each of the five Carbowax 20M modified supports evaluated both with and without OV-210 coating. Fig. 1a–d illustrate typical chromatograms obtained for monocrotophos on four of the five non-coated Carbowax 20M modified supports.

When comparing the five reconditioned supports it was found that the most desirable chromatographic behavior was obtained with the Carbowax 20M modified Gas-Chrom P and Q. Modified Chromosorb G was also good with the exception that peaks were generally broader, indicating lower column efficiency. Chromosorb W modified with Carbowax 20M was not as good as was originally anticipated. More tailing was observed for many of the compounds tested and peaks were broad, indicating poor column efficiency. Some of the pesticides also gave additional large peaks indicating thermal breakdown of the parent compound. Coating the modified supports with OV-210 generally improved the chromatographic behavior of the pesticides. Figs. 1b and 2 depict a typical improvement using the OV-210 coated support on Chromosorb W. Monocrotophos still tailed some but a significant improvement over the non-coated Chromosorb W was noted.

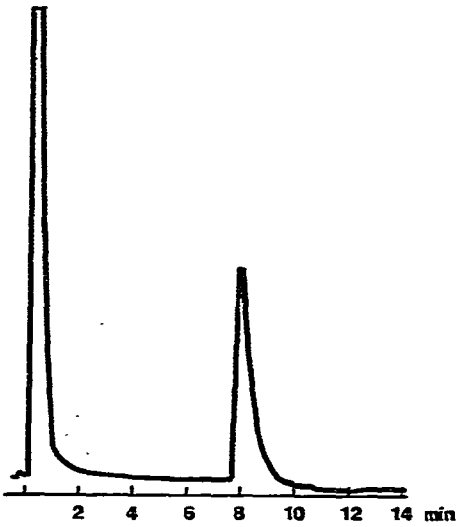

Fig. 2. Chromatogram of monocrotophos on Carbowax 20M-modified Chromosorb W coated with 5% OV-210. Column temperature, 200°.

Separation of the pesticides, as expressed by retention times relative to parathion, was generally comparable for the deactivated supports, with the exception of Chromosorb P (Table IV). However, resolution of the pesticide was not always as good as might be desired even though there was an improvement over most published reports, particularly with lindane, diazinon, atrazine and simazine. Coating the supports with OV-210 changed the *RRT* for many of the compounds as well as improved the separations not possible with the deactivated support alone (Table V).

When comparing *RRT* values and chromatographic behavior expressed by peak shape with coated OV-210 non-treated and coated OV-210 deactivated supports,

there was considerable improvement in favor of the coated deactivated supports (Fig. 3a–h). Most of the *RRT* values of the pesticides were similar between the coated deactivated supports while this was not always true for the non-treated supports. Commercially prepared packings including a 3% OV-210 on Chromosorb W (Pierce, Rockford, Ill., U.S.A.) and a 10% OV-210 on Gas-Chrom Q (Applied Science Labs.) were also compared in this portion of the study (Fig. 3c and Table III). These materials were used as received from the supplier after conditioning for 24 h at 225°. With few exceptions, the deactivated supports coated with OV-210 were superior to the non-treated supports for those chemicals tested (Fig. 3a–h).

The results of this study indicate the importance of a well deactivated solid support when attempting GC separation of certain pesticides. The use of silylating reagents has traditionally provided a relatively inert support surface, but this work indicates that for some specific applications, Carbowax 20M modified supports offer significant advantages.

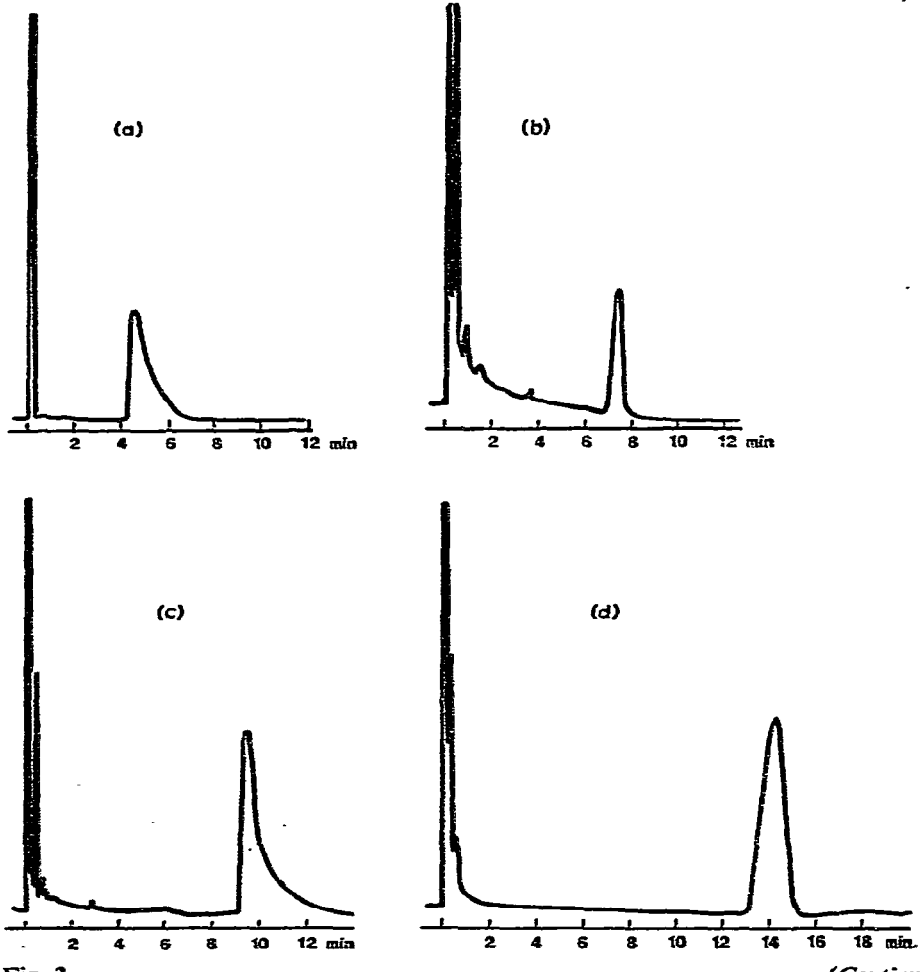

Fig. 3.

(Continued on p. 420)

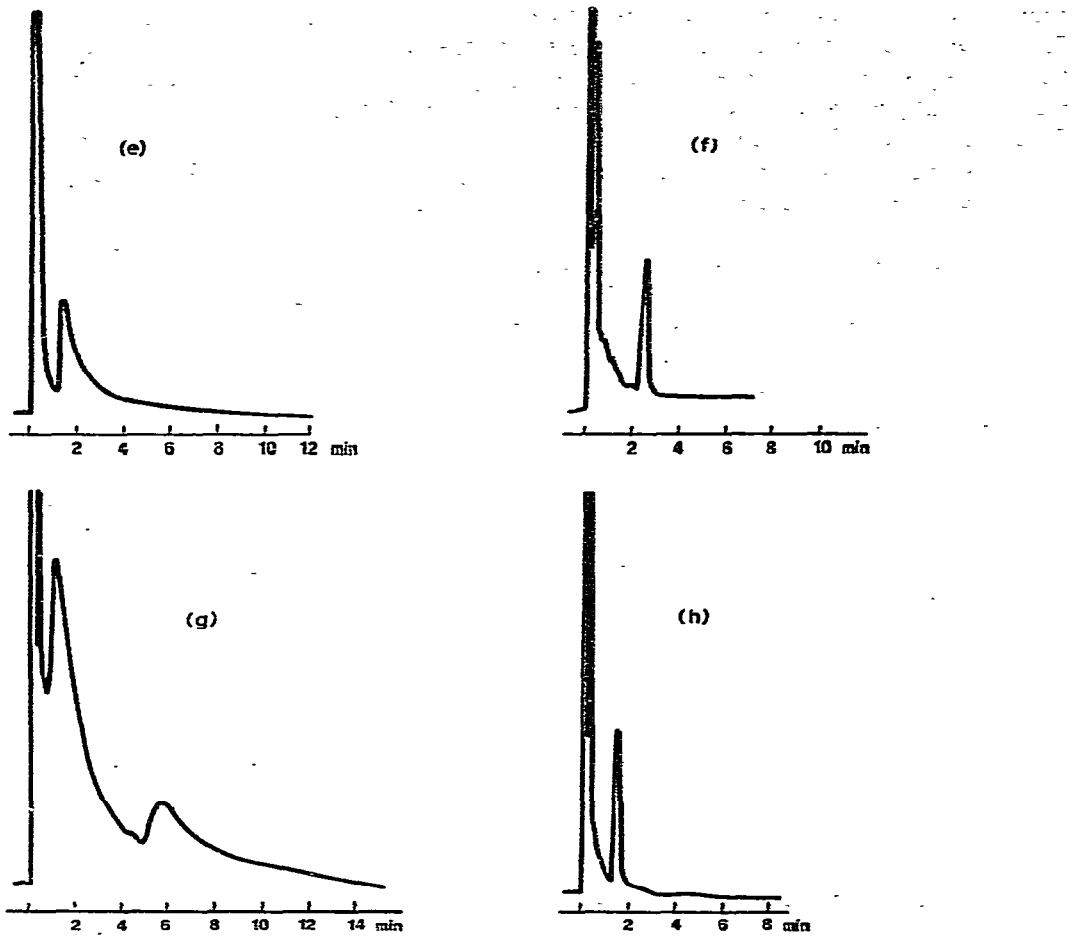

Fig. 3. Chromatogram of disulfoton (a) on non-treated Chromosorb P coated with 10% OV-210 (column temperature, 200°); (b) on Carbowax 20M-modified Chromosorb coated with 10% OV-210 (column temperature, 200°). (c) Chromatogram of mevinphos on non-treated Chromosorb G coated with 10% OV-210 (column temperature, 200°). (d) Mevinphos on Carbowax 20M-modified Chromosorb G coated with 10% OV-210 (column temperature, 200°). (e) Mevinphos on non-treated Chromosorb W HP coated with 3% OV-210 (column temperature, 175°). (f) Mevinphos on Carbowax 20M-modified Chromosorb W coated with 5% OV-210 (column temperature, 175°). (g) Mevinphos on non-treated Gas-Chrom Q coated with 5% OV-210 (column temperature, 175°). (h) Mevinphos on Carbowax 20M-modified Gas-Chrom Q, coated with 5% OV-210 (column temperature, 175°).

Where optimum separations of various compounds have been attempted in the past there have been two basic chromatographic considerations, one dealing with the stationary phase and the other with the solid support. By deactivating a support one can reduce the first factor to a minimum. The characteristics attributed to the stationary phase can then serve as the primary consideration for separating various compounds.

It should be added that since this work was completed there are now many suppliers that can provide such deactivated supports commercially. However, these

materials are very expensive and it might be advantageous for some laboratories to prepare their own modified supports following the procedure and precautions described.

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