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Received for review October 4, 1988. Accepted March 6, 1989.

Recoveries of Chlorinated Hydrocarbon Pesticides from Fat Using Florisil and Silica Sep-Paks

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Florisil and silica Sep-Paks were evaluated as a means of separating chlorinated hydrocarbon pesticides from fat; GC with electron-capture detection was used to measure residues spiked at the 0.005–0.5-μg level into 0.1 g of soybean oil. Fourteen single-peak residues and two multipeak residues, technical chlordane and Arochlor 1260, were tested. Results with several solvents showed that 1% diethyl ether in hexane used with the Florisil Sep-Pak gave the best overall recovery. Interferences in the electron-capture chromatograms are discussed.

Sep-Pak cartridges and other commercially available disposable columns offer several advantages over open-column chromatography for the analyst: speed of separation, low volume of solvent required, small amount of sample needed, and ability to separate or concentrate trace levels of compounds (Winterlin et al., 1981; Gorder and Dahm, 1981). Both bulk Florisil (Luke et al., 1984; Stein and Narang, 1984) and Florisil cartridges have been used to separate a variety of organic pollutants, including PCB (Lerman et al., 1982), PBB (Hu et al., 1982), the chlorinated hydrocarbons HCB and aldrin (Chiang, et al., 1987), and PCP and its derivatives (Mundy and Machin, 1981). In addition, silica cartridges have been used to separate PCB cogeners in oil (Steichen et al., 1982), carbofuran in soil (Gorder and Dahm, 1981), and aldicarb in potato extracts (Cochrane and Lanouette, 1981). As many countries, including the United States, still allow free or restricted use of some chlorinated pesticides, it is of interest to search for simpler multiresidue methods than existing ones (EPA, 1980). In most studies of pollutant recoveries from fat, the subject of separation efficiency is usually not discussed, and where it is, the fat passing into the sample eluate has been measured gravimetrically (Ansari and Hendrix, 1985). However, the use of Sep-Pak cartridges to separate phospholipids from neutral lipids and fatty acids has been reported (Hamilton and Comai, 1984).

In the present study we tested the recovery of 16 chlorinated pesticides, including 14 single-peak residues and two multipeak residues, technical chlordane and Arochlor 1260 PCB, using a variety of solvents on both Florisil and silica Sep-Paks. The regime used is rapid and involves only prerinsing, sample loading, elution, evaporation, reconstitution, and injection. The first five steps require about 20 min. In addition, the efficiency of each cleanup was measured by hydrolyzing the fat passing into the Sep-Pak eluate into methyl esters and then chromatographing and summing the areas of the hydrolysate peaks. Partially hydrogenated soybean oil was chosen for recovery studies because it is a uniform fat, containing >99% triglyceride, <0.1% moisture, and <0.1% free fatty acid content. Furthermore, the fatty acid composition of soybean oil is relatively simple, with typically >99% of fatty acids in the C_{16}–C_{18} range.

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MATERIALS AND METHODS

Apparatus. Perkin-Elmer Sigma 4 gas chromatographs with Ni^{63} electron capture detectors were used for pesticide recoveries. Ancillary equipment included a Perkin-Elmer AS-100B autosampler, a Dynatech GC-311H autosampler, and 1-mV recorders. The glass column used was 2 m × 4 mm (i.d.) and packed with 3.5% SE30/5.25% OV210 on 100/120-mesh Gas Chrom Q (custom packed). Columns used for confirmation were a 2 m × 4 mm (i.d.) column packed with 1.5% SP-2250/1.95% SP-2401 on 100/120-mesh Supelcoport (Supelco, Inc., Bellefonte, PA); a 2 m × 4 mm (i.d.) column packed with 3% SP-2100 on 100/120-mesh Supelcoport (Supelco, Inc.); and a 30 m × 0.32 mm (i.d.) DB-5 capillary column with 1-μm film thickness (J & W Scientific, Folsom, CA). Injector and detector temperatures for all pesticide columns were 300 °C. Oven temperatures were 200 °C for the SE30/OV210, 225 °C for the SP-2250/SP-2401, and 195 °C for the SP-2100 and DB-5 columns. Nitrogen at 30 cm^3/min was used for both carrier and makeup with packed columns. Carrier flow for the DB-5 was 1 cm^3/min of hydrogen with 30 cm^3/min nitrogen makeup to the detector. DB-5 was operated in the split mode at a split ratio of 1:20.

A Hewlett-Packard 5890 GC with FID was used for fatty acid methyl ester (FAME) profiles. Ancillary equipment was a Hewlett-Packard 3393A integrator and 7673 A autosampler. The column used was a 30 m × 0.53 mm (i.d.) DB-225 column at 1 μm film thickness (J & W Scientific). Carrier and makeup gas flow rates were 6 and 25 cm^3/min helium, respectively. Temperatures: injector, 200 °C; detector, 220 °C; column, 180 °C for 13 min, to 210 °C at 5 °C/min, then hold for 20 min.

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Table I. Standard Pesticide Solutions

standard	concn, μg/mL	RRT[a]
HCB	0.005	0.42
α-BHC	0.010	0.50
lindane	0.010	0.62
heptachlor	0.010	0.82
aldrin	0.015	1
HE	0.015	1.58
TNC	0.020	1.75
DDE	0.025	2.0
dieldrin	0.025	2.42
endrin	0.040	2.82
DDD	0.040	3.08
DDT	0.050	3.60
methoxychlor	0.100	5.45
mirex	0.035	4.90
chlordane	0.11	6 peaks
PCB 1260	0.54	16 peaks

[a] Retention time relative to aldrin on the SE 30/OV210 column.

Reagents. All pesticide standards were obtained from the EPA (Pesticides and Industrial Chemicals Repository, MD-8, Research Triangle Park, NC). These include hexachlorobenzene (HCB), α-BHC, γ-BHC (lindane), heptachlor, aldrin, heptachlor epoxide (HE), *trans*-nonachlor (TNC), *p,p'*-DDE, dieldrin, endrin, *p,p'*-DDD, *p,p'*-DDT, mirex, methoxychlor, technical chlordane, and Arochlor 1260 PCB. Stock solutions of each compound were prepared in isooctane and diluted to intermediate standards in hexane (pesticide grade, Fisher Scientific). The 14 single-peak residues were combined into a working standard (Table I). Chlordane and PCB standards were prepared separately.

FAME standards were purchased from Supelco: C 12:0, C 13:0, C 14:0, C 14:1, C 15:0, C 16:0, C 16:1, C 17:0, C 18:0, C 18:1, C 18:2, C 18:3, C 19:0, C 20:0, C 20:1, C 20:4, C 21:0, C 22:0, C 22:1, C 22:6, C 24:0. Standards were dissolved in benzene and combined into a single mixture with concentrations ranging from about 0.01 to 0.5 mg/mL. All pesticide and FAME standards were stored at −20 °C.

PROCEDURE

Pesticide Recoveries. Florisil and silica Sep-Paks (Waters Associates, Milford, MA) were prerinsed with 10 mL of the same solvent used for elution. Soybean oil (0.1 g) was dissolved in either 1 mL of pesticide standard or hexane and the resultant mixture applied to the cartridge with a glass syringe. Without allowing the Sep-Pak to dry, the sample was eluted with either 10 or 20 mL of eluting solvent. Solvent was evaporated in a 35 °C water bath under nitrogen and the residue dissolved in 1 mL of hexane.

Fat Recoveries. Sep-Pak eluates were collected as above and dissolved in 1 mL of benzene after evaporation. In a 5-mL reaction vial was added 2.5 mL of BF_3–methanol (Supelco) and the sample incubated at 105 °C for 15 min. After cooling, 1 mL of water and 1 mL of benzene were added, the mixture was shaken and allowed to settle, and the benzene phase was taken for injection. With each set of samples a control of 0.1 g of soybean oil was hydrolyzed.

RESULTS AND DISCUSSION

The results of a linearity check for the fat hydrolysis method are shown in Figure 1. Fatty acid peaks in the sample were identified and summed to a total peak area. Response is linear from 0.01 to 0.11 g of oil hydrolyzed, with a regression equation of $y = 1777\,x + 2.77$, with total peak area as the dependent variable. The R^2 value was 0.998. These results show that fat recoveries in Sep-Pak eluates can be quantitated with 0.1 g of oil. This method is faster than the AOAC method (1984), which requires a larger sample size; although we have used 0.1 g of oil for these studies, an equivalent amount of oil could be loaded by dissolving larger sample weight in more solvent and taking an aliquot.

Pesticide and fat recoveries from Florisil using four eluting solvents are shown in Table II. Recoveries were run three to five times each, and the average and standard deviation (in parentheses) are given; peak height was used

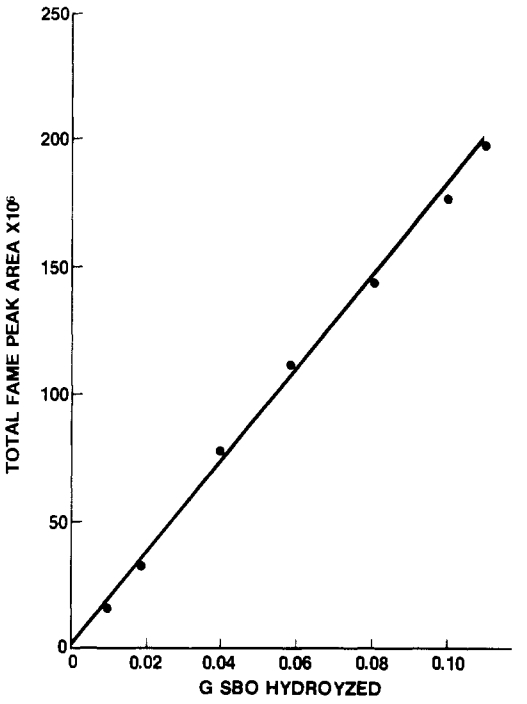

Figure 1. Fat hydrolysis check of soybean oil with BF_3–methanol.

Table II. Florisil Sep-Pak Recoveries with Various Eluting Solvents

	10 mL of hexane	20 mL of hexane	10 mL of 1% Et_2O in hexane	10 mL of 2% Et_2O in hexane
fat passed	0.43 (0.54)	1.1 (0.58)	0.66 (0.11)	7.0 (2.4)
pesticide				
HCB	80 (7.5)	101 (9.0)	92 (7.6)	88 (13.6)
α-BHC	80 (5.9)	95 (2.2)	86 (10.3)	83 (15.4)
lindane	61 (25.5)	98 (3.7)	80 (16.6)	79 (11.6)
heptachlor	111 (12.7)	133 (12.1)	136 (21.0)	113 (6.5)
aldrin	92 (3.9)	100 (9.0)	98 (4.2)	89 (10.1)
HE	10 (9.8)	69 (2.2)	93 (8.5)	89 (10.1)
TNC	90 (7.8)	90 (9.5)	94 (6.5)	89 (12.8)
DDE	96 (3.8)	103 (3.3)	95 (4.1)	90 (13.3)
dieldrin	2 (1.9)	45 (7.4)	62 (13.0)	59 (7.4)
endrin	2 (2.6)	47 (6.9)	72 (7.8)	69 (6.2)
DDD	64 (21.4)	106 (5.1)	106 (11.9)	96 (8.6)
DDT	92 (3.5)	101 (4.5)	104 (9.5)	96 (10.1)
methoxychlor	0.6 (1.1)	33 (8.0)	58 (26.0)	79 (3.7)
mirex	97 (5.0)	104 (1.7)	99 (2.6)	94 (11.3)
chlordane	96 (3.1)			
PCB 1260	96 (5.7)			

for pesticide quantitation. Using 10 mL of hexane, elution gives quantitative recoveries for DDE, mirex, chlordane, and PCB 1260. The PCB result is in agreement with the results of Lerman et al. (1982), and all the peaks in the chromatograms of chlordane and PCB were counted for quantitation. Increasing elution volume to 20 mL of hexane allows HCB, BHC, lindane, aldrin, DDD, and DDT to be quantitatively recovered, while recoveries for HE, dieldrin, endrin, and methoxychlor are increased significantly. Recoveries for these four compounds are further enhanced by increasing solvent polarity by addition of diethyl ether (Et_2O) (MCB Chemicals, Norwood, OH). The only advantage of 2% diethyl ether in eluting solvent was an increase in methoxychlor recovery. Fat retention by the Florisil Sep-Pak was >98% except for the highest polarity solvent, where it was >90%. Quantitation of heptachlor on the SE 30/OV 210 column is not possible because of an interference, as shown in Figure 2. However, this interference can be eliminated and the pesticide peaks confirmed by injection onto other GC columns. Results from a single cleanup injected onto four different columns

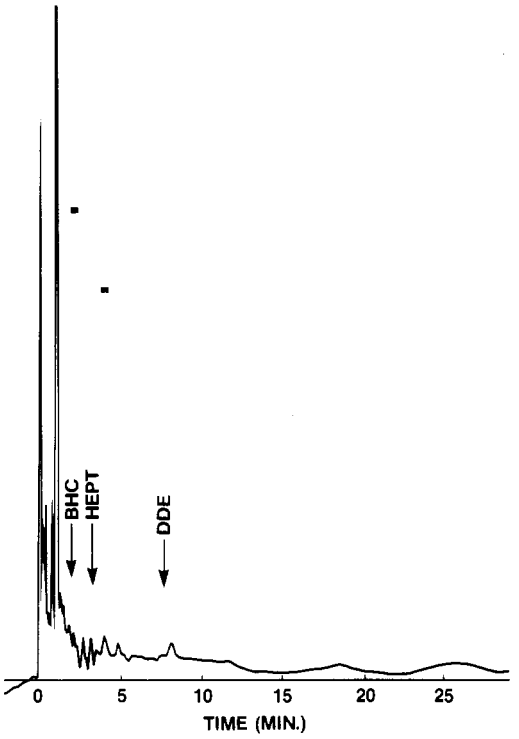

Figure 2. Electron-capture chromatogram of a Florisil eluate with soybean oil on the SE 30/OV 210 column. Elution with 10 mL of diethyl ether in hexane. Peak heights for BHC and heptachlor are shown.

Table III. Confirmation of Peak Identity by Injection on Four Columns (Elution with 1% Et_2O in Hexane from Florisil)

	% recovery			
pesticide	SE30/OV210[a]	SP2250/SP2401	SP-2100	DB-5
HCB	81	86	98	98
BHC	76	87	74	81
lindane	58	64	98	74
heptachlor	110	92	102	101
aldrin	92	93	100	98
HE	81	76	85	103
TNC	86	96	102	88
DDE	96	97		
dieldrin	49	49	80[b]	80[b]
endrin	66	63	69	76
DDD	92	95	97	101
DDT	93	86	100	95
methoxychlor	22	19	20	26
mirex	95	94	98	104

[a] Column used for recoveries in Table II. [b] Compounds not resolved.

Table IV. Interferences in a Reagent Blank Containing Soybean Oil (Elution with 1% Et_2O in Hexane from Florisil)

column	pesticide	peak ht (interfer/std)
SE30/OV210	heptachlor	0.10
SP2250/SP2401	BHC	0.15
SP2100	HCB	0.32
	lindane	0.35
DB-5	HE	0.36

are shown in Table III, with general agreement between columns. Some of the discrepancy in recovery can be explained by comparison with interferences of a blank, shown in Table IV. On the SE 30/OV 210 column the heptachlor interference is 10% of the peak height of the standard, which accounts for the high heptachlor recovery

Table V. Silica Sep-Pak Recoveries with 10 mL of Eluting Solvent

	PE	1% Et_2O in PE	2% Et_2O in PE
fat passed	0.07 (0.09)	1.1 (1.4)	21.8 (6.6)
pesticide			
HCB	67 (10.7)	107 (1.7)	106 (13.0)
BHC	44 (5.9)	99 (3.9)	100 (8.2)
lindane	4.2 (7.4)	91 (7.8)	96 (7.8)
heptachlor	84 (11.3)	122 (13.8)	129 (15.0)
aldrin	86 (7.5)	108 (1.7)	108 (8.6)
HE	0	107 (8.2)	105 (9.4)
TNC	84 (10.9)	99 (4.5)	104 (8.7)
DDE	93 (6.6)	116 (11.5)	111 (10.0)
dieldrin	0	75 (5.8)	88 (4.6)
endrin	0	100 (15.5)	97 (5.4)
DDD	54 (14.7)	116 (7.1)	112 (8.8)
DDT	94 (10.7)	115 (1.5)	109 (10.3)
methoxychlor	0	42 (12.0)	87 (8.3)
mirex	98	115 (6.5)	108 (11.2)
chlordane	89 (10.7)	104 (7.6)	
PCB 1260	102 (4.7)	98 (7.4)	

Table VI. Silica Sep-Pak Recoveries with 20 mL of Eluting Solvent

	PE	1% Et_2O in PE
fat passed	0.58 (0.52)	24.8[a]
pesticide		
HCB	83 (12.1)	100[a]
BHC	80 (6.6)	97
lindane	68 (1.2)	103
heptachlor	106 (11.5)	127
aldrin	93 (8.2)	107
HE	38 (5.7)	102
TNC	88 (8.6)	99
DDE	99 (3.3)	108
dieldrin	17 (12.6)	107
endrin	18 (14.0)	110
DDD	100 (6.1)	118
DDT	101 (4.2)	109
methoxychlor	3.3 (4.7)	118
mirex	96 (5.7)	107
chlordane	100[a]	
PCB 1260	116[a]	

[a] Single determination only.

in pesticide eluate in Table III. Similarly there is a BHC interference on the SP 2250/SP 2401 column, HCB and lindanes interference on the SP-2100 column, and a HE interference on the DB-5 column, which also show up as high recoveries in the pesticide eluate. When these are taken into account, between-column differences are minimal.

Table V shows recoveries with a silica Sep-Pak eluted with petroleum ether (Fisher Scientific, Fairlawn, NJ). Mirex and PCB 1260 are quantitatively recovered with 10 mL of petroleum ether, but if 1% diethyl ether is added, then HCB, BHC, aldrin, HE, TNC, DDE, endrin, DDD, DDT, and chlordane are recovered. Addition of 2% diethyl ether gives recovery of lindane, although the fat in the eluate has increased significantly. With this eluting solvent only dieldrin and methoxychlor are not totally recovered. Table VI shows recoveries with 20 mL of petroleum ether from silica Sep-Paks. Good recoveries are seen with DDE, DDD, DDT, mirex, chlordane, and PCB 1260. Increasing polarity with 1% diethyl ether allows recovery of all compounds but at the expense of a large amount of fat in the eluate.

The results of the study indicate that certain of the chlorinated hydrocarbon pesticides can be separated from fat with use of silica and Florisil cartridges. Of the eluting

Table VII. Binding of Feed-Grade Fats to Florisil Sep-Pak (Elution with 10 mL of 1% Et_2O in Hexane)

	load 1	load 2	load 3	load 4
moisture content, %	0.96	0.04	0.08	0.62
free fatty acid content, %	53.0	3.2	2.9	49.8
% fat in eluate (std dev)	2.5 (1.7)	5.8 (0.78)		

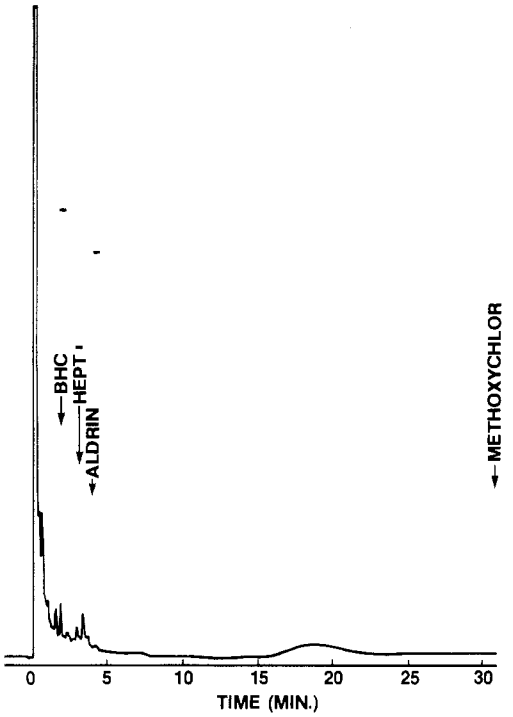

Figure 3. Electron-capture chromatogram of a Florisil eluate of soybean oil.

solvents tested, 1% diethyl ether in hexane gave the best overall pesticide recovery with maximum fat retention on Florisil; similarly, 1% diethyl ether in petroleum ether worked best with the silica cartridge. Since a single sample can be run in less than 30 min, this method may be an alternative to other multiresidue methods used for fats. It is noted that all the standard peaks present in chlordane and PCB 1260 were seen in the Florisil and silica eluates. Quantitation of the oil or fat passing into the eluate will allow other workers to measure the efficiency of cleanup using other eluting solvents and other commercially available cartridges.

Although the fat-binding properties of both Florisil and silica are excellent with soybean oil and some of the eluting solvents used for this study, other types of oil or fat may exhibit different degrees of binding. Table VII lists fat in eluates using two loads of feed-grade fat blended into poultry feed. In addition to being variable in moisture and free fatty acid content, these products may contain fat from any vegetable or animal source and are frequently mixtures of several species of oil or fat. Furthermore, they may contain significant amounts of unsaponifiable matter and have been subjected to distillation, hydrolysis, bleaching, and treatment with antioxidants. As can be seen, more fat is eluted from both loads compared to soybean oil. Further work is indicated to tell whether these differences are due to the species of fat involved or to their processing.

Chromatograms of two other loads of feed-grade fat from Florisil are shown in Figures 4 and 5 with a chromatogram of soybean oil injected on the same column, the SP2250/SP2401, for comparison (Figure 3). Both chromatograms are free of interferences, and the BHC interference seen from soybean oil is absent in these fats.

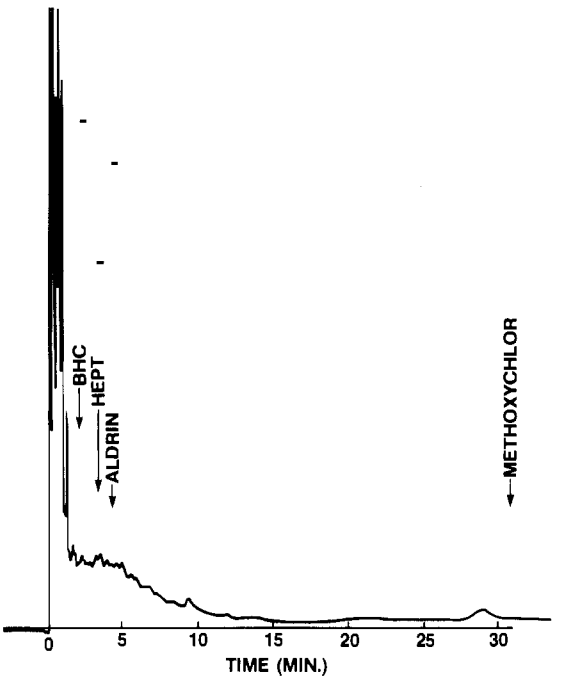

Figure 4. Electron-capture chromatogram of a Florisil eluate of feed fat, load 3.

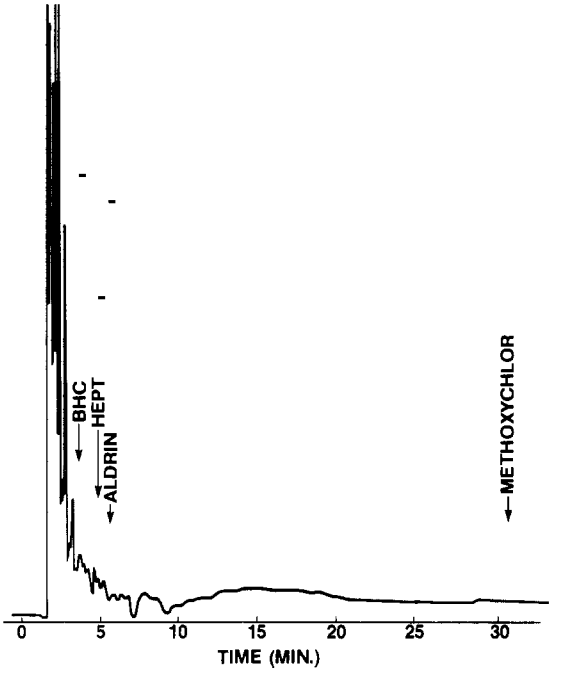

Figure 5. Electron-capture chromatogram of a Florisil eluate of feed fat, load 4.

Pesticide recovery should thus be applicable to these fats as well as soybean oil.

ACKNOWLEDGMENT

Assistance from Georgia Butler and Lena Hendrix with some Sep-Pak cleanups is appreciated.

Registry No. HCB, 118-74-1; α-BHC, 319-84-6; HE, 1024-57-3; TNC, 39765-80-5; DDE, 72-55-9; DDD, 72-54-8; DDT, 50-29-3; florisil, 1343-88-0; lindane, 58-89-9; heptachlor, 76-44-8; aldrin, 309-00-2; dieldrin, 60-57-1; endrin, 72-20-8; methoxychlor, 72-43-5; mirex, 2385-85-5; chlordane, 12789-03-6; arochlor 1260, 11096-82-5.

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Received for review April 1, 1988. Revised manuscript received October 20, 1988. Accepted April 14, 1989.

Nonpolar Lipids of *Amaranthus palmeri* S. Wats. 1. Fatty Alcohols and Wax Esters (Saturated)

Oliver D. Dailey, Jr.,* Ray F. Severson, and Richard F. Arrendale

In order to isolate and identify the nonpolar lipids of *Amaranthus palmeri*, a common agronomically significant weed, the ground leaves and flowering parts of dried plants were extracted with hexanes. The extract was subjected to column chromatography, thin-layer chromatography, and finally capillary GC/MS studies. In this paper, the compositions of the saturated fatty alcohol and wax ester components will be discussed. These classes of compounds have not been characterized in any prior studies of *Amaranthus* species. The wax ester isolate consisted of a series of C_{36}–C_{56} homologues, with the C_{40}, C_{42}, C_{44}, C_{46}, and C_{48} homologues predominating. The major wax ester fatty acids were C_{16}, C_{18}, C_{20}, C_{22}, and C_{24}. Similar trends in carbon number distribution were found between free and bound fatty alcohols, with the C_{22}, C_{24}, C_{26}, C_{28}, C_{30}, and C_{32} homologues predominating.

Palmer amaranth (*Amaranthus palmeri* S. Wats) is a common agronomically significant weed whose soil-incorporated residues have been observed to inhibit the growth of certain crop plants, most notably carrot and onion. The weed residues are also autotoxic. (Menges, 1987).

Laboratory seed germination bioassays of crude organic solvent extracts of *A. palmeri* plant parts (including those discussed in this paper) indicated the presence of both promotive and inhibitory compounds (Bradow, 1985). Aqueous extracts of the leaves and thyrses (flowering parts) had no significant effect on any of the seeds tested (Bradow, 1985). In their studies of *A. palmeri*, Fischer and Quijano (1985) isolated phytol and chondrillasterol from a petroleum ether extract of aerial parts and fatty acids, chondrillasterol, 3-methoxy-4-hydroxynitrobenzene, vanillin, and 2,6-dimethoxybenzoquinone from a dichloromethane extract of ground roots.

There have been a number of investigations of the composition of the seed lipids of both vegetable amaranths [*Amaranthus caudatus* (*Amaranthus edulis*), *Amaranthus cruentus*, *Amaranthus dubius*, *Amaranthus tricolor* (*Amaranthus gangeticus*)] and weedy species (*Amaranthus hybridus*, *Amaranthus retroflexus*, *Amaranthus spinosus*) (Becker et al., 1981; Dixit and Varma, 1971; Fernando and Bean, 1985; Bressani et al., 1987; Lorenz and Hwang, 1985; Opute, 1979; Stoller and Weber, 1970). These studies dealt primarily with fatty acid composition.

Lakshminarayana et al. (1984) published a comprehensive report of the lipid and fatty acid composition of the leaves of young *A. gangeticus* (*A. tricolor*) plants. The nonpolar lipids consisted of pigments, hydrocarbons, sterols, ester waxes, fatty acid methyl esters, triglycerides, diglycerides, monoglycerides, and fatty acids. The fatty acid composition of the ester waxes in decreasing order was palmitic, linolenic, stearic, oleic, linoleic, and lauric.

The studies described in this paper were initiated as part of a research effort to isolate and identify allelopathic compounds of *A. palmeri*. In screening for potential al-

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