

**Table I. Color Reaction of Rotenone and Related Compounds with Hydriodic Acid Reagent**

Compound <sup>a</sup>	Color Produced	
	Within 0.5 hr.	After 6 hr.
Rotenone	Blue	Blue
Elliptone	Pink to violet	Violet to purple
Isorotenol	None	Light tan
Deguclin	None	Light tan
Dihydrodeguclin	None	Light tan
Dehydrodeguclin	None	Light tan
Tephrosin	None	Light tan
Toxicarol	None	Light tan
Sumatrol	None	Faint blue

<sup>a</sup> Tested at 50  $\mu\text{g}$ . per sq. cm. on Whatman No. 1 filter paper.

after spraying and persists for several days to several weeks under humid conditions. Should a more permanent record be desired, the chromatogram may be stored in a vacuum desiccator over sodium hydroxide pellets. As little as 4  $\mu\text{g}$ . per sq. cm. can be detected.

The rotenoids tested are listed in Table I. Only elliptone gave an immediate reaction with the spray reagent. Unlike rotenone, it gave a pink or violet color which slowly changed in hue with time. Rotenone and elliptone were readily separated by the chromatographic technique described. Rotenone gave an  $R_f$  of 0.88 and elliptone an  $R_f$  of 0.80 (measuring to the front of the band). Even when the two zones

partially overlapped, the initial colors were sufficiently distinctive to permit the boundaries of the zones to be determined.

As a further test of specificity, the reagent was sprayed on paper chromatograms of the unpurified acetone extracts from *D. elliptica* roots and *T. vogelii* leaves. Both chromatograms showed a single blue band, which was in the position of rotenone. A pink band due to elliptone was found with *D. elliptica* but was absent with *T. vogelii*. Elliptone has been found in *D. elliptica* (7) but not in *T. vogelii* (4, 16). Except for rotenone and elliptone, none of the other materials present in the total extracts of these two plants produced color with spray reagent.

Although the search for interfering compounds has not been exhaustive, rotenone is the only compound which has been found to give a characteristic blue color with hydriodic acid.

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## INSECTICIDE PERSISTENCE AND TRANSLOCATION

### Residues of Aldrin and Heptachlor in Soils and Their Translocation into Various Crops

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THE application of pesticides directly to soil and the contamination of soils through "fallout" after crop treatments cause concern over the potential accumulation of toxic residues in soils. It is essential, therefore, to determine at what rate and frequency a given chemical can be applied to soils without gradual accumulation of pesticidal residues over a period of years. Under certain conditions residues of some insecticides can be translocated from soils into crops (2, 3, 5, 6). This makes it necessary to determine at what concentrations of pesticidal residues in soils and under what conditions no residues would be absorbed by plants grown therein. It should also be known at what concentration of pesticides in soils translocated residues would still be within the limits of tolerances set by the Food and Drug Administration.

To obtain some insight into these problems, a study was initiated in 1958 at the University of Wisconsin whereby soils were treated at abnormally high rates of aldrin and heptachlor. Two years later this study was expanded to include application rates comparable with those normally used in agriculture. To investigate possible translocation of insecticidal residues, various crops were grown on the contaminated soils.

#### Procedure

**Soil Treatments at Abnormally High Dosages and Soil Sampling.** In May 1958, duplicated 30- $\times$ -40-foot Carrington silt loam plots were treated (3) with emulsions of aldrin and heptachlor at 5 or 25 pounds per acre. The soils were rototilled to a depth of 4 to 5 inches. After that a 30- $\times$ -16-foot portion of each plot was seeded with alfalfa.

Only soils to which the insecticides were applied at 5 pounds per 5-inch acre were re-treated at the same rate each May from 1959 through 1962. Since the dense alfalfa cover was maintained throughout the experiment, the re-treated areas were reduced to 30  $\times$  24 feet. At the end of the 5 years, all plots had been treated with a total of 25 pounds of insecticide per 5-inch acre (5  $\times$  5 or 25 pounds).

For soil residue studies, the intention was to determine if and to what extent the insecticides would accumulate in the soil following yearly applications of 5 pounds per 5-inch acre; how fast they would disappear after being applied at one massive dosage; what the residue levels would be at the end of 5 years in the differently treated plots; and finally, what influence a dense cover crop would have on the persistence of these residues in soils.

Loam soils were treated with aldrin and heptachlor in five yearly dosages of 5 pounds per acre, one massive dose of 25 pounds per acre, or at "normal" rates of 1, 2, or 3 pounds per acre per year over a 3-year period. Various crops were grown on the insecticide-treated soils. The insecticidal applications in five yearly 5-pound dosages resulted in 1.7 to 2 times higher soil residues than in soils treated only once at 25 pounds per acre. Repeated applications resulted, after 3 or 5 years, in residue levels close to 20% of the total applied dosages. A dense cover crop of alfalfa prolonged the persistence of the insecticidal residues in soils. Certain crops grown in these soils did not absorb measurable amounts of insecticidal residues, while others translocated the chemicals in various amounts. Carrots absorbed more insecticidal residues than any other crop tested. Some quantitative relationship between soil residue levels and translocated insecticides was noticed. Potatoes, radishes, and carrots, grown on soil treated with aldrin at 1 pound per acre, either contained no measurable amounts of residues or residues at concentrations of 0.03 and 0.05 p.p.m., respectively. In general, more residues were found in crops grown on heptachlor-treated soils than in those from aldrin-contaminated soil plots.

Six-inch soil samples were collected as described (3) immediately after treatment in 1958 and in August (harvest time) and October of each year. A final soil sample was also collected in May 1963.

**Crop Growth and Crop Sampling.** Various crops were grown each year in 30-foot rows on the treated and untreated plots. The crops grown in 1958 and 1959 as well as the analytical results have been reported (3). Carrots (Red Cored Chantenay), potatoes (Russet Sebago), beets (Detroit Dark Red), radishes (Early Scarlet Globe), and cucumbers (Straight Eight) were grown every year. Lettuce (Great Lakes), turnips (Purple Top Strap Leaf), and cabbage (Globe, Yellow Resistant) were grown in 1960, broccoli (Green Sprouting) and celery (Summer Pascal) in 1961, and parsnips (All American) and celery (Summer Pascal) in 1962.

To eliminate the sampling problem, all the edible parts of a particular crop grown in one 30-foot row were harvested and brought into the laboratory for processing. Each part was brushed in warm water to remove adhering soil particles, then rinsed with acetone from a wash bottle, and immediately rinsed with warm water. The clean crop parts were macerated with a food grinder (Hobart, Model T-215 food cutter), the ground material was mixed, and aliquots were placed in plastic bags within 1-quart ice cream cartons, frozen, and held until extraction. Only the inner leaves of lettuce were used. They were washed with water and then macerated. Cabbage was macerated after removal of the outer leaves.

**Soil Treatments at Practical Levels and Soil Sampling.** The rate of disappearance or possible accumulation of aldrin and heptachlor residues in soils treated at practical levels was studied for 3 years. In May 1960, the insecticides were applied as an emulsion to Carrington silt loam plots as described (3) at 1, 2, or 3 pounds per 5-inch acre. After each plot had been rototilled, an area of 30 × 16 feet was seeded with alfalfa. The area of each plot which was not covered with alfalfa (30 × 24

feet) was re-treated in May 1961 and 1962 at 1, 2, or 3 pounds per 5-inch acre. In this way, a total of 3 (3 × 1), 6 (3 × 2), or 9 (3 × 3) pounds per 5-inch acre of aldrin or heptachlor was applied over the 3-year period.

Six-inch soil samples were collected as described (3) in August and October of each year and also in May 1963, at the end of the 3-year period.

**Crop Growth and Crop Sampling.** Carrots (Red Cored Chantenay), potatoes (Russet Sebago), radishes (Early Scarlet Globe), and beets (Detroit Dark Red) were grown in 30-foot rows on the insecticide-treated plots. They were harvested and processed as described.

**Analytical Methods.** During the five years of the experiment, several analytical methods were employed. Soil samples collected in 1960, 1961, and 1962 were analyzed colorimetrically and, in 1963,

by gas-liquid chromatography. Crops grown in 1960 and 1961 were analyzed by colorimetric and bioassay methods. Gas-liquid chromatography was used with crop samples obtained in 1962.

The extraction, cleanup, and colorimetric analyses of soils and crops were performed as previously described (3). In addition, purified crop extracts were bioassayed, using vinegar flies (*Drosophila melanogaster* Meig.) as the test insect (3). Ascending paper chromatography (9) was used with fractions of extracts containing aldrin, dieldrin, heptachlor, or heptachlor epoxide residues. When insecticides were detected colorimetrically and by bioassay, positive results were also obtained by paper chromatography.  $R_f$  values secured with purified crop extracts were identical with  $R_f$  values obtained with reference grade insecticide.

Crops in 1962 and soils in 1963 were

**Table I. Recoveries of Aldrin (A), Dieldrin (D), Heptachlor (H), and Heptachlor Epoxide (HO) Residues from Carrington Silt Loam Soils Treated with Aldrin or Heptachlor**

(Anal. method, gas-liquid chromatography)

	Insecticides Applied, Lb./5-Inch Acre				
	Aldrin		Heptachlor		
	5 × 5 <sup>a</sup>	25 <sup>b</sup>	5 × 5 <sup>a</sup>	25 <sup>b</sup>	
	Recovered, Lb./6-Inch Acre in May 1963 <sup>c</sup>				
Open <sup>d</sup>					
A + D	4.69 ± 0.0	2.78 ± 0.20	H + HO	4.42 ± 0.41	2.14 ± 0.01
% D <sup>e</sup>	66	97	% HO <sup>f</sup>	21	62
% appl. <sup>g</sup>	19	11	% Appl.	18	9
	5 <sup>h</sup>	25 <sup>b</sup>		5 <sup>h</sup>	25 <sup>b</sup>
Alfalfa <sup>i</sup>					
A + D	1.51 ± 0.19	6.59 ± 0.10	H + HO	1.20 ± 0.13	7.26 ± 0.30
% D	98	95	% HO	92	71
% appl.	30	26	% appl.	24	29

<sup>a</sup> Aldrin or heptachlor applied at 5 lb./5-inch acre in May of each year (1958, 1959, 1960, 1961, 1962). Total application: 25 lb./acre over 5-year period.

<sup>b</sup> Aldrin or heptachlor applied at 25 lb./5-inch acre only in May 1958.

<sup>c</sup> Average of results from replicated experimental plots.

<sup>d</sup> "Open" part of plot in which various crops were grown in rows 2 to 3 feet apart.

<sup>e</sup> Dieldrin in per cent of total residues recovered (A + D).

<sup>f</sup> Heptachlor epoxide in per cent of total residues recovered (H + HO).

<sup>g</sup> Residues recovered in per cent of applied insecticidal dosages.

<sup>h</sup> Aldrin or heptachlor applied at 5 lb./5-inch acre only in May 1958.

<sup>i</sup> Area of plot seeded in alfalfa after insecticidal treatment in 1958.

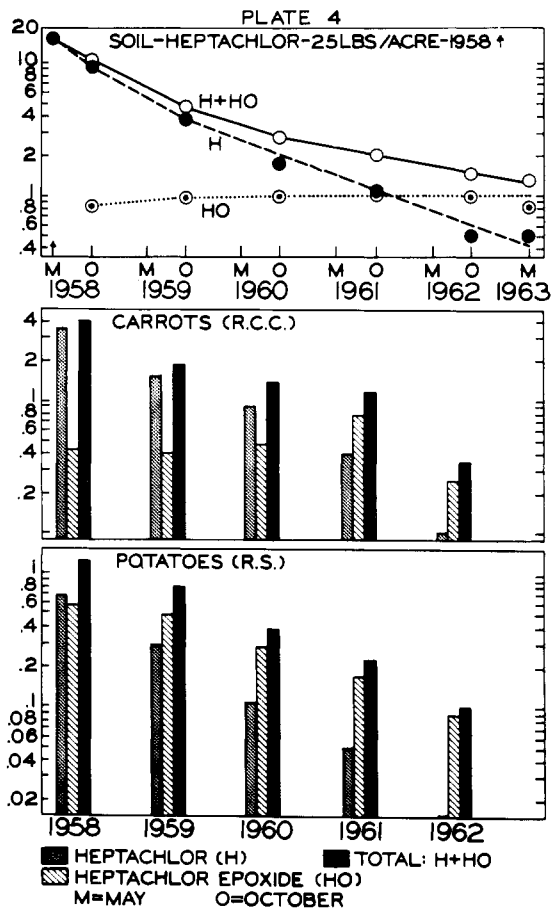
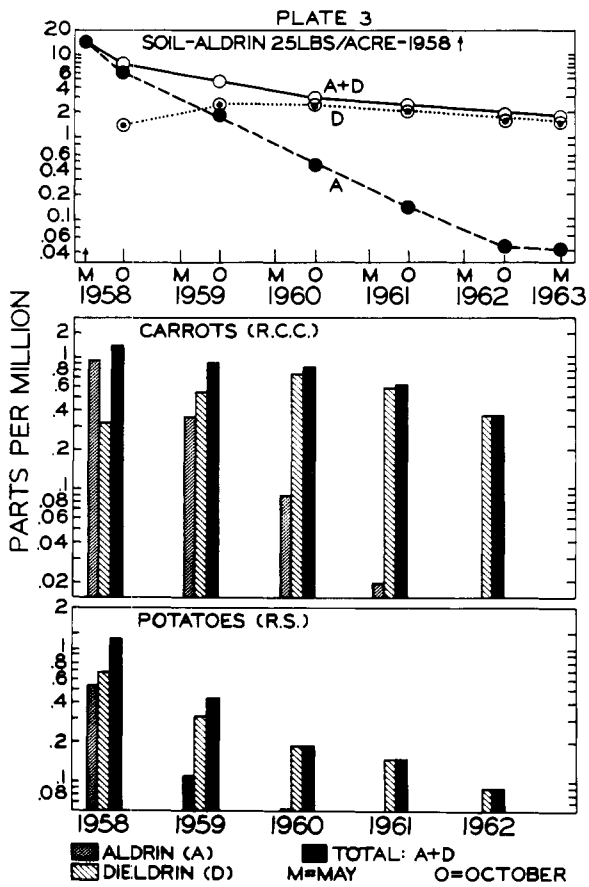
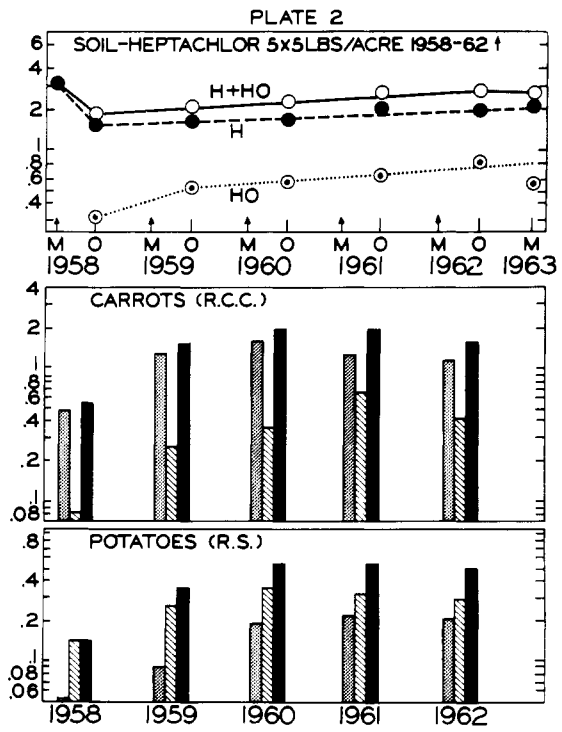
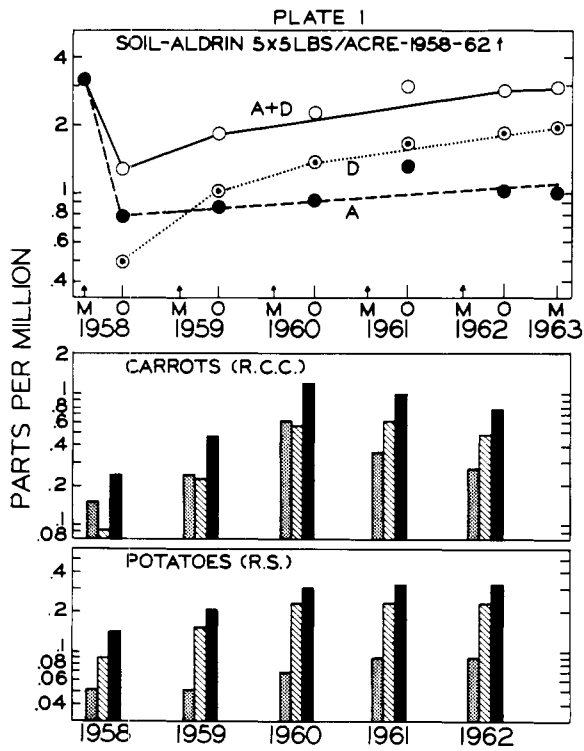


Figure 1. Recoveries of insecticidal residues from Carrington silt loam and from crops grown in these soils

Plates 1 and 2: Aldrin or heptachlor applied (†) at 5 pounds per 5-inch acre in May 1958, 1959, 1960, 1961, and 1962

Plates 3 and 4: Aldrin or heptachlor applied (†) at one dosage of 25 pounds per 5-inch acre in 1958

**Table II. Recoveries of Aldrin (A) and Dieldrin (D) Residues from Carrington Silt Loam Soils and Crops, Grown during 1960, 1961, and 1962 on Aldrin-Treated Plots**

	Aldrin Applied to Soil, Lb./5-Inch Acre											
	5 <sup>a</sup>					25 <sup>b</sup>						
	Recovered from Soils at Harvest Time, P.P.M. <sup>c</sup>											
	1960		1961		1962		1960		1961		1962	
A + D	% D <sup>d</sup>	A + D	% D	A + D	% D	A + D	% D	A + D	% D	A + D	% D	
	2.30	45	2.90	51	3.52	57	2.52	78	2.14	93	1.95	95
Recovered from Crops at Harvest, P.P.M.												
Beets												
C <sup>e</sup>	0.13	100	0.19	89	...	...	0.14	100	0.10	90	...	...
B <sup>e</sup>	0.12	100	...	...	...	...	0.14	100	...	...	...	...
G <sup>f</sup>	...	...	...	...	0.34	94	...	...	...	...	0.12	100
Radish												
C	0.15	66	0.23	70	...	...	0.10	100	0.14	100	...	...
B	0.14	86	...	...	...	...	0.11	100	...	...	...	...
G	...	...	...	...	0.64	80	...	...	...	...	0.25	100
Cucumber												
C	0.11	100	0.16	87	...	...	0.12	100	0.17	100	...	...
B	0.11	100	0.12	100	...	...	0.10	100	0.16	100	...	...
G	...	...	...	...	0.11	91	...	...	...	...	0.16	100
Lettuce												
C	0.03	100	...	...	...	...	0.04	100	...	...	...	...
B	0.04	100	...	...	...	...	0.04	100	...	...	...	...
Turnip												
C	0.05	100	...	...	...	...	0.09	100	...	...	...	...
B	0.04	100	...	...	...	...	0.06	100	...	...	...	...
Cabbage												
C	g	g	...	...	...	...	g	g	...	...	...	...
B	g	g	...	...	...	...	g	g	...	...	...	...
Broccoli												
C	g	g	g	g	...	...	g	g	g	g	...	...
B	g	g	g	g	...	...	g	g	g	g	...	...
Celery												
C	...	...	0.04	100	...	...	...	...	0.02	100	...	...
B	...	...	g	g	...	...	g	g	g	g	...	...
G	...	...	...	...	0.01	100	...	...	...	...	...	...
Parsnips												
G	...	...	...	...	0.68	75	...	...	...	...	0.51	100

<sup>a</sup> Aldrin applied at 5 lb./5-inch acre in May of each year (1958 through 1962). Total application: 25 lb./acre over 5-year period.  
<sup>b</sup> Aldrin applied at 25 lb./5-inch acre in May 1958 only. <sup>c</sup> Colorimetric analyses. <sup>d</sup> Dieldrin in per cent of total residue recovered (A + D). <sup>e</sup> Bioassay. <sup>f</sup> Gas-liquid chromatography. <sup>g</sup> No measurable amounts of residues.

extracted as previously described (5) and analyzed by gas-liquid chromatography. A Jarrell-Ash gas chromatograph, Model 700, equipped with a 100-mc. tritium electron affinity detector was used. A 4-foot (1.22-meter) column (<sup>3</sup>/<sub>16</sub>-inch, 4.76-mm. inside diameter) containing 80- to 90-mesh Anakrom ABS (acid, alcoholic base, washed and siliconized) and 1% SE 30-neopentyl glycol adipate (terminated) was conditioned before use for 48 hours at 200° C. For aldrin and dieldrin analyses a column pressure of 25 p.s.i. of nitrogen, giving a flow rate of 100 ml. per minute, was employed. The injector temperature was held at 250° C., the column temperature at 150° C., and the detector cell at 208° C. The detector potential was 15 volts. Added amounts of aldrin and dieldrin to crop material were recovered to an extent of 90 to 98%. When analyses for heptachlor residues were performed, the column temperature was held at 129° C. A column pressure of 25 p.s.i. of nitrogen resulted in a flow rate of 110 ml. per minute. Temperatures of the injector and the detector cell were as described. The detector potential, however, was 11 volts. Added amounts of heptachlor or heptachlor epoxide to

the crop material were recovered to an extent of 95 to 99%.

### Results and Discussion

**Soil and Crop Residues Following Treatment of Carrington Silt Loam at Abnormally High Dosages of Aldrin and Heptachlor.** After aldrin had been applied to the loam soils at 5 pounds per 5-inch acre in 1958, a relatively rapid decline of the residues occurred in the soil during the first summer (Figure 1, plate 1). However the amounts of aldrin and dieldrin recovered from the soil in the fall were still 41% (1.28 p.p.m.) of the applied dosage. With annual re-treatments a slight but steady increase in insecticidal soil residues occurred. At the end of 5 years (May 1963) a residue concentration (aldrin and dieldrin) of 2.93 p.p.m. or 4.69 pounds per 6-inch acre remained in the soil (Table I). This represented 19% of the total insecticide applied (5 × 5 pounds) or 94% of a single yearly dosage of 5 pounds per acre.

The amounts of dieldrin formed from aldrin in the soil increased with time. Its concentration in the soil was equal to

that of aldrin (2 p.p.m.) in the summer of 1959, whereas after 5 years it represented 66% of the recovered residues.

The sections of the plots which were seeded with alfalfa in 1958 were treated only once at 5 pounds per 5-inch acre. Five years later, close to one third of the applied dosage was still present in the soil, principally in the form of dieldrin (Table I).

The application of aldrin at one massive dosage of 25 pounds per 5-inch acre resulted in considerably lower residue levels in soils after 5 years (Figure 1, plate 3) when compared to the levels in plots where aldrin was applied in five 5-pound increments. The most rapid loss occurred during the first summer, when 50% of the applied chemical disappeared. Thereafter, the rate of decline of the residues was slower, leaving a residue in the soil which amounted to 1.74 p.p.m. or 2.78 pounds per 6-inch acre in May 1963. Eighty-nine per cent of the applied insecticide disappeared in 5 years. What was left was practically all in the form of dieldrin. In fact, 2.5 years after the insecticidal application, more than 85% of the remaining res-

**Table III. Recoveries of Heptachlor (H) and Heptachlor Epoxide (HO) Residues from Carrington Silt Loam Soils and Crops, Grown During 1960, 1961, and 1962 on Heptachlor-Treated Plots**

	Heptachlor Applied to Soil, Lb./5-Inch Acre											
	5 <sup>a</sup>						25 <sup>b</sup>					
	Recovered from Soils at Harvest, P.P.M. <sup>c</sup>											
	1960		1961		1962		1960		1961		1962	
H + HO	% HO <sup>d</sup>	H + HO	% HO	H + HO	% HO	H + HO	% HO	H + HO	% HO	H + HO	% HO	
	2.75	23	3.03	22	4.04	24	3.66	31	2.34	50	1.60	63
Recovered from Crops at Harvest, P.P.M.												
Beets												
C <sup>e</sup>	0.26	73	0.24	67	...		0.21	86	0.09	100	...	
B <sup>e</sup>	0.27	74	...		...		0.19	84	...		...	
G <sup>f</sup>	...		...		0.39	69	...		...		0.08	92
Radish												
C	0.27	63	0.37	60	...		0.15	94	0.16	88	...	
B	0.29	59	...		...		0.16	88	...		...	
G	...		...		0.37	57	...		...		0.11	91
Cucumber												
C	0.05	100	0.17	65	...		0.05	100	0.12	83	...	
B	0.07	86	...		...		0.07	86	...		...	
G	...		...		0.09	78	...		...		0.08	87
Lettuce												
C	0.03	100					g					
B	0.03	66					0.01	100				
Turnip												
C	0.06	100					0.09	100				
B	...						...					
Cabbage												
C	0.02	100					0.02	100				
B	0.02	100					0.03	100				
Broccoli												
C			g						g			
B			g						g			
Celery												
C			g						g			
B			0.03	33	...				g			
G			...		0.01	100			...			
Parsnips												
G					1.53	21					0.73	72

<sup>a</sup> Heptachlor applied at 5 lb./5-inch acre in May of each year (1958 through 1962). Total application: 25 lb./acre over 5-year period.  
<sup>b</sup> Heptachlor applied at 25 lb./5-inch acre in May 1958 only. <sup>c</sup> Colorimetric analyses. <sup>d</sup> Heptachlor epoxide in per cent of total residues recovered (H + HO). <sup>e</sup> Bioassays. <sup>f</sup> Gas-liquid chromatography. <sup>g</sup> No measurable amounts of residues.

idues were dieldrin. Covering the soil with alfalfa lengthened persistence of the residues 2.3 times (Table I).

Heptachlor as such was more persistent than aldrin, because the rate of epoxidation of heptachlor was much slower than the rate at which aldrin was converted into dieldrin. However, the total residues of heptachlor and heptachlor epoxide recovered from soils were very similar to those found in the aldrin-treated plots (Figure 1 and Table I). In soils to which heptachlor had been applied at 5 pounds per acre annually for 5 years, the amount of heptachlor epoxide never exceeded 29% of the totally recovered residues. This did occur, however, in soils treated once with 25 pounds per 5-inch acre, although it took 3½ years (fall 1961) until equal concentrations (1 p.p.m.) of heptachlor and heptachlor epoxide were reached. Covering a part of the plot with alfalfa increased the persistence of the heptachlor residues by a factor of 3.3 (Table I).

Certain crops grown in these soils did not absorb measurable amounts of residues, while other crops translocated

the chemicals to various degrees. A quantitative and qualitative relationship between soil residue levels and translocated insecticides was noticed especially with carrots and potatoes (Figure 1).

Although the concentrations of the insecticidal residues in the soils treated with 5 × 5 pounds per acre increased slightly over the years, the concentrations of translocated residues in both carrots and potatoes increased only from 1958 to 1959. After that the concentrations within potatoes were either similar or decreased slightly, as in carrots.

Carrots translocated more residues than any of the other crops. However, they contained relatively (in per cent of the totally recovered residues) less dieldrin or heptachlor epoxide than potatoes and most other crops. Dieldrin in carrots ranged from 37 to 64% (1958-1962) of the totally recovered residues, while the amount of heptachlor epoxide was from 14 to 27% of all toxicants recovered. In potatoes, however, these figures were 64 to 73% dieldrin and 60 to 100% heptachlor epoxide. The largest amounts of total residues were found in carrots and

potatoes grown on heptachlor-treated soils.

The decreasing residue levels in soils treated once in 1958 with 25 pounds per 5-inch acre resulted in a decline in the amounts of residues translocated into crops (Figure 1, plates 3 and 4) more pronounced with potatoes than with carrots. However, the relative amounts of dieldrin or heptachlor epoxide (in per cent of the totally recovered residues) increased over the years. The recovered residue was all in the form of dieldrin with potatoes in the third year of the experiment and with carrots during the fifth year. On the heptachlor-treated soils, comparatively less epoxide was found in the crops. Over 50% of the total residue recovered from potatoes was in the form of heptachlor epoxide in the second year. This occurred with carrots only in the fourth year.

Results obtained with all the other crops are summarized in Tables II and III. Broccoli from aldrin- or heptachlor-treated plots contained no measurable amounts of insecticidal residues. Cabbage contained small amounts of heptachlor epoxide, but no measurable

**Table IV. Aldrin (A), Dieldrin (D), Heptachlor (H), and Heptachlor Epoxide (HO) Residues Recovered from Carrington Silt Loam Soils, after Treatment with Aldrin or Heptachlor in May of 1960, 1961, and 1962**

		Insecticides Applied, Lb./5-Inch Acre						
		Aldrin			Heptachlor			
		1 <sup>a</sup>	2 <sup>a</sup>	3 <sup>a</sup>	1 <sup>a</sup>	2 <sup>a</sup>	3 <sup>a</sup>	
		Recovered, Lb./6-Inch Acre <sup>b</sup>						
1960, October	A + D	0.27	0.69	1.06	H + HO	0.26	0.54	1.04
	% D <sup>c</sup>	41	35	32	% HO <sup>d</sup>	25	18	12
	% appl. <sup>e</sup>	27	35	35	% appl.	26	27	36
1961, October	A + D	0.54	1.10	1.68	H + HO	0.43	1.26	2.02
	% D	68	55	49	% HO	26	19	13
	% appl.	27	27	28	% appl.	21	31	34
1962, October	A + D	0.66	1.18	1.89	H + HO	0.59	1.41	2.51
	% D	73	70	70	% HO	30	25	18
	% appl.	22	20	21	% appl.	20	23	28
1963, May	A + D	0.59	1.41	1.93	H + HO	0.61	1.25	1.97
	% D	79	68	69	% HO	27	24	18
	% appl.	20	23	22	% appl.	20	21	22
Alfalfa <sup>f</sup> covered	A + D	0.22	0.76	0.67	H + HO	0.22	0.59	0.81
	% D	100	97	96	% HO	83	74	68
	% appl.	22	38	22	% appl.	22	30	27

<sup>a</sup> Insecticides applied at 1, 2, or 3 lb./5-inch acre in May of each year (1960, 1961, 1962). Total application: 3, 6, or 9 lb./acre over 3-year period.

<sup>b</sup> By colorimetric analyses in 1960, 1961, and 1962; by gas-liquid chromatography in 1963.

<sup>c</sup> Dieldrin in per cent of total residues recovered (A + D).

<sup>d</sup> Heptachlor epoxide in per cent of total residues recovered (H + HO).

<sup>e</sup> Recovered residues in per cent of totally applied insecticide.

<sup>f</sup> Treated at 1, 2, or 3 lb./5-inch acre in 1960 only.

amounts of aldrin or dieldrin. The amounts of translocated residues in lettuce, turnips, and celery ranged from less than 0.01 to 0.09 p.p.m. Parsnips, which are closely related to carrots, represented the other extreme and contained excessively high amounts of insecticides. In all cases where toxicants were found in crops, dieldrin or heptachlor epoxide was the major constituent of the totally recovered residues.

**Soil and Crop Residues Following Treatment of Carrington Silt Loam**

**at Practical Levels of Aldrin or Heptachlor.** Soil residue data from plots to which aldrin or heptachlor had been applied at practical dosages of 1, 2, and 3 pounds per 5-inch acre indicated a general trend in the persistence of these insecticides in the soil (Table IV). The residue level in all soils, 3 years after the first insecticidal application, was 20 to 23% of the total amount of the applied insecticides. Similar data were obtained with soils treated for 5 years with the insecticides at 5 pounds

per 5-inch acre per year. The absolute amount of residues increased slowly in all plots and ranged from 59 to 69% of the yearly dosages of 1, 2, or 3 pounds per acre when determined in May 1963. Concentrations of the residues in the aldrin and heptachlor-treated plots were very similar. Relatively more dieldrin (in per cent of totally recovered residues) than heptachlor epoxide was recovered. The alfalfa portion of the treated plots showed residue recoveries of 22 to 38% of the applied dosages in 1963, although the soil had been treated only once in 1960. Also, most of the heptachlor residues were in the form of its epoxide. The prolonged persistence of aldrin and heptachlor residues in densely covered soils was probably due to a reduction in volatilization of the chemicals (7, 4). Those areas had not been cultivated over the years, as was done with the remainder of the plots on which crops had been grown.

Potatoes and radishes grown in 1960 on the soil treated with aldrin at 1 pound per 5-inch acre did not contain measurable residues, but the concentration in carrots was 0.05 p.p.m. (Table V). Even during the next 2 years (1961 and 1962) residue concentrations in crops grown in this soil did not exceed 0.1 p.p.m. Among the crops grown on soil treated with heptachlor at 1 pound per acre per year, only carrots contained residues in excess of 0.1 p.p.m. (Table VI).

Soil treatment with aldrin at 2 pounds per acre resulted in higher residues in crops. However, only carrots (and radishes in 1962) contained residues greater than 0.1 p.p.m. Residue levels in crops grown on the comparably heptachlor-treated soil were slightly

**Table V. Recoveries of Aldrin (A) and Dieldrin (D) Residues from Carrington Silt Loam Soils and Crops, Grown during 1960, 1961, and 1962 on Aldrin-Treated Plots**

		Aldrin Applied to Soil, Lb./5-Inch Acre								
		1 <sup>a</sup>			2 <sup>a</sup>			3 <sup>a</sup>		
		1960	1961	1962	1960	1961	1962	1960	1961	1962
		Recovered from Soils at Harvest Time, P.P.M. <sup>b</sup>								
A + D		0.15	0.34	0.47	0.35	0.70	0.77	0.67	1.14	1.34
% D <sup>c</sup>		46	50	64	37	48	62	30	39	62
		Recovered from Crops at Harvest, P.P.M.								
Carrots										
A + D		0.05 <sup>bd</sup>	0.09 <sup>bd</sup>	0.08 <sup>e</sup>	0.11 <sup>bd</sup>	0.19 <sup>bd</sup>	0.30 <sup>e</sup>	0.30 <sup>bd</sup>	0.30 <sup>bd</sup>	0.27 <sup>e</sup>
% D		60	55	77	63	58	63	47	50	63
Potatoes										
A + D		<sup>bd</sup> f	0.03 <sup>b</sup>	0.04 <sup>e</sup>	0.05 <sup>bd</sup>	0.10 <sup>b</sup>	0.09 <sup>e</sup>	0.06 <sup>bd</sup>	0.14 <sup>b</sup>	0.11 <sup>e</sup>
% D			100	100	100	80	92	83	79	92
Radishes										
A + D		<0.01 <sup>bd</sup>	0.02 <sup>b</sup>	0.07 <sup>e</sup>	0.02 <sup>bd</sup>	0.07 <sup>b</sup>	0.15 <sup>e</sup>	0.08 <sup>bd</sup>	0.07 <sup>b</sup>	0.18 <sup>e</sup>
% D		100	100	76	100	86	73	63	72	72
Beets										
A + D			0.01 <sup>bd</sup>	0.02 <sup>e</sup>		0.04 <sup>bd</sup>	0.07 <sup>e</sup>		0.05 <sup>bd</sup>	0.14 <sup>e</sup>
% D			100	100		100	100		100	96

<sup>a</sup> Aldrin applied at 1, 2, or 3 lb./5-inch acre in May of each year (1960, 1961, 1962). Total application: 3, 6, or 9 lb./acre over 3-year period

<sup>b</sup> By colorimetric analyses. <sup>c</sup> Dieldrin in per cent of total residues recovered (A + D). <sup>d</sup> By bioassay. <sup>e</sup> By gas-liquid chromatography.

<sup>f</sup> No measurable amount of residues.

**Table VI. Recoveries of Heptachlor (H) and Heptachlor Epoxide (HO) Residues from Carrington Silt Loam Soils and Crops Grown during 1960, 1961, and 1962 on Heptachlor-Treated Plots**

	Heptachlor Applied to Soil, Lb./5-Inch Acre								
	1 <sup>a</sup>			2 <sup>a</sup>			3 <sup>a</sup>		
	1960	1961	1962	1960	1961	1962	1960	1961	1962
	Recovered from Soils at Harvest Time, P.P.M. <sup>b</sup>								
H + HO	0.19	0.32	0.41	0.39	0.67	0.90	0.93	1.25	1.33
% HO <sup>c</sup>	16	19	27	13	16	23	8	11	20
	Recovered from Crops at Harvest, P.P.M.								
Carrots									
H + HO	0.14 <sup>d</sup>	0.18 <sup>d</sup>	0.15 <sup>e</sup>	0.31 <sup>d</sup>	0.36 <sup>d</sup>	0.44 <sup>e</sup>	0.51 <sup>d</sup>	1.12 <sup>d</sup>	0.69 <sup>e</sup>
% HO	21	22	35	15	17	22	14	12	19
Potatoes									
H + HO	0.05 <sup>bd</sup>	0.05 <sup>b</sup>	0.10 <sup>e</sup>	0.10 <sup>bd</sup>	0.13 <sup>b</sup>	0.14 <sup>e</sup>	0.17 <sup>bd</sup>	0.27 <sup>b</sup>	0.27 <sup>e</sup>
% HO	100	100	67	70	77	82	77	63	72
Radishes									
H + HO	0.03 <sup>bd</sup>	0.03 <sup>bd</sup>	0.07 <sup>e</sup>	0.07 <sup>bd</sup>	0.07 <sup>bd</sup>	0.12 <sup>e</sup>	0.09 <sup>bd</sup>	0.10 <sup>bd</sup>	0.21 <sup>e</sup>
% HO	100	100	71	100	70	65	100	80	62
Beets									
H + HO		0.01 <sup>bd</sup>	0.05 <sup>e</sup>		0.05 <sup>bd</sup>	0.10 <sup>e</sup>		0.08 <sup>bd</sup>	0.29 <sup>e</sup>
% HO		100	70		100	70		87	65

<sup>a</sup> Heptachlor applied at 1, 2, or 3 lb./5-inch acre in May of each year (1960, 1961, and 1962). Total application: 3, 6, or 9 lb./acre over 3-year period.

<sup>b</sup> By colorimetric analyses.

<sup>c</sup> Heptachlor epoxide in per cent of total residues recovered (H + HO).

<sup>d</sup> By bioassay.

<sup>e</sup> By gas-liquid chromatography.

higher (Table VI). Most of the crops grown on soils treated with aldrin at 3 pounds per 5-inch acre did not contain relatively higher insecticidal residues.

These results were obtained with a silt loam under Wisconsin conditions (average yearly rainfall, 30.16 inches or 766.07 mm.; average yearly temperature, 45.3° F. or 7.3° C.; average temperature for the period May through October, 62.4° F. or 16.9° C.). In areas of higher temperatures and higher humidities, insecticidal soil residues would dissipate faster (7, 7, 8) and possibly present a reduced hazard in terms of translocation of toxicants from soils into crops.

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## INSECTICIDE STORAGE IN FAT

### Storage of Heptachlor Epoxide in the Body Fat and Its Excretion in Milk of Dairy Cows Fed Heptachlor in Their Diets

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CHLORINATED hydrocarbon insecticides ingested or absorbed by dairy animals may be stored in the animals' body fat and excreted in milk (3-5, 8, 14, 15, 17, 19). Animals grazed on pastures treated with heptachlor (6, 7, 12, 13) and chlordan (18) excreted heptachlor epoxide for a considerable time after treatment and re-

moval from the treated pastures. Dairy cattle fed alfalfa hay grown on soil treated with heptachlor excreted significant quantities of heptachlor epoxide (9). Some heptachlor epoxide was found in the milk up to 45 days after intake was discontinued.

Such compounds vary appreciably in their propensity for storage and excretion

(8). Some, such as aldrin and heptachlor, are not stored as such but are converted to and are stored as their epoxides, dieldrin and heptachlor epoxide. In the case of aldrin, it was apparent that an animal receiving X amount of aldrin daily would store more dieldrin than a comparable animal receiving the same amount of dieldrin