Absorption of Some Chlorinated Hydrocarbon Insecticides from Soils into Various Crops

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A study was made of the extent to which insecticides may be absorbed and translocated from contaminated soils into plant tissues and the relationships among absorbance of insecticidal residues, soil types, and crops. Lindane, DDT, and aldrin were absorbed into crops, the degree being dependent on the crop, the soil type in which the crop had grown, the insecticide, and its concentration within the soil. Carrots not only absorbed more insecticide than any other crop, but in the case of lindane accumulated greater quantities of the chemical than occurred in the soil. The insecticides were most readily absorbed from a sandy loam and least from a muck soil. The amounts absorbed by the same crop from the same type of soil were not in direct proportion to the concentration of the insecticide recovered from the soil, and relatively less insecticide was absorbed from soils in which the insecticide was most concentrated. Crops grown in aldrin-treated soils contained within their tissues both aldrin and dieldrin.

DURING RECENT YEARS most of the research concerning insecticide residues and crops was done with plants, to which insecticides were applied in spray or dust formulations. However, a large percentage of the applied insecticide finds its way into the soil (*2, 6, 9*) and persists there over relatively long periods of time, depending on the soil type and the nature of the chemical. Moreover, insecticides are applied in many cases directly to the soil. Investigations conducted during recent years indicated that insecticides not only persist or accumulate within the soil (*11*) but may change into other toxic compounds (*5, 12*). As soils containing insecticidal residues are used in agriculture, the insecticides might be translocated into various plant parts (*1, 3, 7, 8, 13*) and consumed by humans and animals.

To obtain more information as to the extent to which insecticides are translocated from contaminated soils into plant tissues, and the relationships between the absorbance of insecticidal residues, soil types, and crops, several insecticides (lindane, DDT, and aldrin) were applied in various concentrations to three soil types. Crops were grown, 1, 2, and 3 years after soil application on these plots and in all cases the edible plant parts were analyzed for insecticide content.

Procedure

Soil Treatment and Sampling. In May 1954, a sandy loam, Miami silt loam, and muck soil were treated with DDT at 10, 100, and 1000 pounds per acre, lindane at 1, 10, and 100 pounds per acre, and aldrin at 2, 20, and 200 pounds per acre. The insecticidal application involved thorough mixing of an emulsifiable concentrate with 10 gallons of water, except that DDT (1000 pounds per acre) was applied as an undiluted emulsion concentrate. Experimental plots (50 × 29 feet) with 15-foot-wide buffer strips in between were divided into 10 × 29 foot subplots and 2-gallon quantities of diluted emulsion were spread as equally as possible with a sprinkling can over each subplot.

Immediately after application, each plot was rototilled to a depth of 4 to 5 inches. Soil samples were collected initially and at 1, 1.5, 2, 2.5, 3, and 3.5 years after treatment. Soil sampling was done with a soil auger and 40 cores ($^3/_4$ inch in diameter and 6 inches long) were collected from each plot. The 50 × 29 foot area of each experimental plot was reduced to 42 × 21 feet for sampling, leaving a 4-foot strip between the boundaries of the treated plot and the sampling area. Before each extraction, the total soil sample was screened and then thoroughly mixed on a sheet of paper (30 × 30 inches) by rolling in different directions. Aliquots of the soil under investigation were taken for extraction and water determination.

Crop Growth and Crop Sampling. One year after soil treatment, in 1955, various crops were seeded or planted in 29-foot rows of the lindane-treated as well as untreated plots. The crops grown were carrots (Red Cored Chantenay), potatoes (Russet Sebago), peas (Wilt Resistant Perfection), beans (Processor), cucumbers (Straight Eight), tomatoes (Urbana), and cabbage (Oak View).

Two years after treatment, in 1956, carrots were grown on all insecticide-treated plots as well as the untreated ones. Three years after soil treatment, in 1957, carrots (Red Cored Chantenay), beets (Imperial Detroit Dark Red), cucumbers (Straight Eight), potatoes (Russet Sebago), radishes (Early Scarlet Globe), and rutabaga (Long Island Neckless) were grown on all treated and untreated plots. At the time of harvest the edible parts of each crop were brought into the laboratory for subsequent analysis.

Because the chief objective of this work was to determine the amount of insecticide absorbed into the crop tissue, care was taken to remove adhering soil particles from the crop surface. Each edible plant part was first brushed in warm water, rinsed with acetone by means of a wash bottle, and immediately thereafter rinsed with warm water. The cleaned crop parts were all passed through a food grinder, after which the macerated material was mixed with a spoon. From this ground and mixed crop material, aliquots were taken for extraction, or were frozen, until it was possible to make an extraction.

Analytical Methods

Colorimetric Analyses. When soils were extracted, 400 grams of field moist soil and 200 grams of anhydrous sodium sulfate were placed together in 2-quart wide-mouthed Mason jars. An additional 100 grams of soil were dried for 24 hours at 46° C. to determine the dry weight of the soil.

When crops were extracted, 300- to 400-gram aliquots of the macerated crop material were placed on paper and mixed with an equal amount of anhydrous sodium sulfate. If after one-half hour the mixture did not appear dry, more anhydrous sodium sulfate was added, until dryness was achieved. After that the crop–sodium sulfate mixture was placed in 2-quart Mason jars for extraction.

The extraction solvents were a mixture of benzene and isopropyl alcohol (2 to 1 by volume) for DDT and a mixture of redistilled hexane and isopropyl alcohol (2 to 1 by volume) for aldrin and dieldrin. Two milliliters of solvent were used per gram of wet soil or crop material. A 1-hour head to end tumbling was applied, after which the supernatant liquid was decanted through filter paper and the recovery volume was recorded to be used for calculating the results.

The isopropyl alcohol was then removed by washing the extract twice with water and then three times with a saturated solution of sodium chloride. The alcohol-free phase was dried over anhydrous sodium sulfate. DDT extracts were cleaned up by passing through a column (6 × 1 inch) of aluminum oxide. Only carrot extracts contained some interfering substances, which had to be removed by an additional shaking in a mixture of Nuchar (neutral) and Celite. The cleaned up extracts were then analyzed by the Schechter-Haller method (*16*). Known amounts of DDT added to insecticide-free material resulted in an average recovery of 94%.

Extracts containing aldrin and dieldrin were cleaned up by using a 6-inch Florisil (60 × 100 mesh) column (20 mm. in diameter), which permitted at the same time a separation of the two insecticides. After the extract had been added to the column, aldrin was eluted by adding 200 ml. of redistilled hexane. Dieldrin was then eluted from the column with 700 ml. of a mixture of redistilled hexane and benzene (4 to 1 by volume). The first 150 ml. collected contained colored substances, but no dieldrin. Therefore, only the remaining fraction of 550 ml. was used for the dieldrin analysis. The hexane-benzene solution was then evaporated to dryness and the dieldrin was taken up in 2 ml. of redistilled hexane. Only the aldrin-containing fraction of the carrot extract required an additional cleanup by passing through a column of (8 × 1 inch) of Attasol-Celite.

Aldrin and dieldrin fractions were analyzed according to the phenylazide method (*14*, *15*). Known amounts of aldrin added to insecticide-free material were recovered to an extent of 92 to 98%. Known amounts of dieldrin added to soils or crops were recovered to an extent of 88 to 93%.

Lindane was analyzed according to the Schechter-Hornstein method (*17*). A change of this method (*10*) eliminated a special extraction procedure and permitted the determination of lindane directly in soils and crops. Known amounts of lindane added to soils or crops were recovered at approximately 100%.

Each analysis was run in duplicate. A soil or crop blank was used for the determination of apparent insecticide content. In addition, known amounts of insecticide were added to insecticide-free soil or crop samples. The unknowns, after a correction for apparent insecticide, were calculated on the basis of the values obtained for the known amounts. Results were finally expressed in parts per million, based on the dry weight for soils and the fresh weight for plants.

Bioassay. For bioassaying soils or crops, *Drosophila melanogaster* Meig. was used as a test insect. The flies were exposed directly to soils (*4*) and crops, which eliminated a special extraction procedure. All aldrin-treated soils as well as the crops grown on those soils—though containing aldrin and dieldrin at the time of the analysis—were measured against an aldrin standard.

Table I. Recoveries of Lindane from Soils and Crops Grown on Plots Treated with Lindane

(Colorimetric analyses)

Lindane	Sandy Loam		Miami Silt Loam		Muck	
	Applied to Soil in 1954, Lb./5 Inch Acre					
	10	100	10	100	10	100
	Recovered from Soil, P.P.M.					
	1955					
Spring	3.04	32.5	2.11	30.8	12.00	123.1
Fall	1.82	25.4	1.49	22.2	8.73	107.0
	1956					
Spring	1.70	24.2	1.33	21.2	8.50	108.0
Fall	0.96	17.5	0.90	13.0	6.66	81.0
	Recovered from Crops, P.P.M.					
Carrots 1955	[a]		13.90	44.65	1.46	19.90
1956	5.99	23.80	2.41	25.40	0.40	11.50
Potatoes 1955	0.62	[a]	0.29	3.61	0.14	3.37
Pea, vines	1.52	18.40	Lost	4.92	0.11	0.79
pods	No pods available		Lost	0.27	0.09	0.10
Cabbage	0.37	2.37	0.00	0.31	0.09	0.15
Cucumbers	No fruit	[a]	0.31	[a]	0.17	0.53
Beans, pods	No pods available		0.07	1.38	0.00	0.23
Tomatoes	0.00	0.11	0.00	0.21	0.00	0.20

[a] Crops did not germinate or died at an early stage.

Table II. Recoveries of Lindane from Soils and from Crops Grown on Plots Treated with Lindane 3 Years Previously

(Colorimetric analyses)

	Sandy Loam			Miami Silt Loam		
	Lindane Applied to Soil in 1954, Lb./5 Inch Acre					
	1	10	100	1	10	100
	Lindane Recovered, P.P.M. in 1957					
	From soils					
Spring	0.00	0.81	16.05	0.00	0.88	13.87
Fall	0.00	0.63	12.10	0.00	0.60	12.57
	From crops					
Carrots	0.00	4.45	35.90	0.16	6.18	32.20
Beets	[a]	[a]	[a]	0.12	0.15	1.50
Cucumbers	[a]	[a]	[a]	0.00	[a]	0.88
Potatoes	0.00	0.06	8.00	0.00	0.05	2.07
Radishes	[a]	[a]	7.00	0.00	0.00	1.48
Rutabaga	0.00	[a]	7.48	0.00	0.34	1.60

[a] Crops did not germinate or died at an early stage.

Results and Discussion

Tables I to V summarize the recoveries of lindane, DDT, aldrin, and dieldrin as found in three soil types, 1, 2, and 3 years after treatment. The amount of insecticides determined within various crops, grown in these soils, is also presented.

One year after soil treatments with lindane, crops were grown on the plots; all the crops had lindane within their tissues, the amount being dependent on the crop, the residue level within the soil, and the soil type (Table I). Carrots not only absorbed lindane from the soil more readily than the other plants investigated, but in some cases "accumulated" lindane within the edible part. When grown on a Miami silt loam, which had been treated with lindane at a rate of 10 pounds per acre one year previously 7.7 times more insecticide was found within the edible part of the carrots (13.9 p.p.m.) than in the soil (1.8 p.p.m. average during the growing season). But only 1.7 times more lindane was found in

carrots grown on a loam that had been treated at a rate of 100 pounds per acre. Another loam plot treated in 1954 with lindane at a rate of 1.0 pound per acre contained no lindane in 1957. Yet carrots and beets grown during the summer of 1957 in this soil contained measurable amounts of lindane when analyzed in the fall of 1957 (Table II). All the other crops contained less lindane (in p.p.m.) than the soils, though differences in insecticide absorption could be noticed between various crops as well as between different parts of the same crop.

Tomato fruits contained the smallest amount of lindane, when compared to the other crops. Peas grown in a Miami silt loam contained 18 times more lindane in their vines than in their pods, and eight times more, when the peas were grown in a muck soil.

The soil type itself seems—in most cases—to have a remarkable influence on the absorption of lindane into crops. During the first year of the experiment, lindane, applied at a rate of 100 pounds per 6-inch acre, was highly phytotoxic on a sandy loam, less on a Miami silt loam, and almost nontoxic in a muck soil. Moreover, the insecticide persisted longer in a muck soil than in a soil of low organic content. Apparently the chemical is adsorbed to the organic matter of the soil to such an extent, that in a muck soil no phytotoxicity was noticeable and the breakdown of the insecticide was slowed down. Even LD_{50} values for insects exposed to soils are considerably higher in a muck soil than in a loam or sandy loam (*4*). It is also possible that the insecticides are dissolved in the organic matter of a muck soil, and, therefore, are less available for metabolism, or pickup by plants.

The binding of insecticides to the organic matter in soils might explain why most of the lindane was found in crops grown in a sandy loam (Table I) and least in crops grown in a muck soil. Carrots grown in 1956 on three soil types—which had been treated with lindane at a rate of 10 pounds per acre 2 years earlier—contained 6 p.p.m. of lindane when grown in a sandy loam, 2.4 p.p.m. in a Miami silt loam, and 0.4 p.p.m. in a muck soil, though the muck contained approximately six times more lindane than the other two soil types. Only in carrots grown in a muck soil, the amount of lindane (in p.p.m.) was smaller than the amount found in the soil.

Potatoes (unpeeled tubers), grown in 1955 on plots treated one year earlier with lindane at a rate of 10 pounds per acre, contained 0.62 p.p.m. when grown in a sandy loam, 0.29 p.p.m. in a Miami silt loam, and 0.14 p.p.m. in a muck soil, though the muck soil contained approximately five times more lindane (8.73 p.p.m.) than the two other soil types (1.82 and 1.49 p.p.m., respectively) when analyzed in the fall of 1955. Two years later, in 1957, potatoes were found (Table II) to contain 8.00 p.p.m. of lindane in tubers grown in a sandy loam treated at a rate of 100 pounds per acre in 1954 and 2.07 p.p.m. in tubers grown in a Miami silt loam treated at a rate of 100 pounds per acre in 1954. Both soil types contained very similar amounts (12.1 and 12.6 p.p.m., respectively) of lindane in the fall of 1957.

Part of the lindane applied to soils breaks down within 2 weeks to a nontoxic compound (*12*), which will still be detected by the colorimetric analysis for lindane, but not by bioassay. The discrepancy between the chemical analyses and the bioassays will be greater, the more the original insecticide has broken down into a nontoxic compound which still responds to the Schechter-Hornstein method (*17*). By using both analytical methods for crops, some information should be obtained as to whether or not the amount of insecticide found within the plant tissues by chemical analyses is the original toxicant. In a sandy loam the quantity estimated by bioassay amounted to 46% of that obtained by chemical analyses, 3.5 years after soil treatment. In a Miami silt loam, the recoveries obtained—3.5 years after treatment—by bioassay amount to 28 and 51% of the estimates secured by chemical analyses.

In carrots the quantities estimated by bioassays amounted to 87.5 and 105% (grown in a sandy loam), and to 96.5 and 57.5% (grown in a Miami silt loam) of those obtained by chemical analyses.

In beets, grown during 1957 on lindane-treated loam plots, the quantities estimated by bioassays amounted to 126 and 85% of those obtained by chemical analyses, and in radishes to 90%. However, so far, not enough crop data are

Table III. Recoveries of DDT and Aldrin from Soils and from Carrots Grown on Soils Treated with DDT and Aldrin 2 Years Previously

	Sandy Loam			*Miami Silt Loam*			*Muck*		
	DDT Applied to Soil in 1954, Lb./5 Inch Acre								
	10	100	1000	10	100	1000	10	100	1000
	DDT Recovered, P.P.M., in 1956								
	From soils[a]								
Spring	2.15	27.0	312.0	2.14	35.6	403.0	6.45	82.3	793.0
Fall	1.65	23.1	261.0	1.95	32.3	322.0	5.60	74.1	775.0
	From carrots[a]								
No carrots	2.45	23.7		0.00	2.37	3.32	0.00	0.00	1.07
	Aldrin Applied to Soil in 1954, Lb./5 Inch Acre								
	2	20	200	2	20	200	2	20	200
	Aldrin Recovered, P.P.M., in 1956								
	From soils[a]								
Spring	0.00	0.24	22.25	0.00	0.69	16.50	0.27	4.94	158.0
Fall	0.00	0.08	12.10	0.00	0.38	12.22	0.19	3.11	100.0
	From carrots[b]								
	0.15	0.71	3.50	0.00	1.19	5.90	0.00	0.32	6.50

[a] Determined by colorimetric analyses. [b] Determined by bioassay.

Table IV. Recoveries of DDT from Soils and Crops Grown on Plots Treated with DDT 3 Years Previously

(Colorimetric analyses)

	Sandy Loam			*Miami Silt Loam*		
	DDT Applied to Soil in 1954, Lb./5 Inch Acre					
	10	100	1000	10	100	1000
	DDT Recovered, P.P.M., in 1957					
	From soils					
Spring	2.18	24.8	261.0	2.01	31.8	335.0
Fall	1.54	18.5	238.0	1.62	24.0	302.0
	From crops					
Carrots	0.32	3.17	10.9	Traces	2.39	10.10
Beets	[a]	[a]	[a]	0.00	[a]	1.35
Cucumbers	[a]	[a]	[a]	0.00	0.00	0.00
Potatoes	Not available	1.63	7.50	Traces	0.92	3.90
Radishes	[a]	[a]	[a]	0.00	0.87	4.34
Rutabaga	Traces	0.89	3.82	0.00	0.33	1.22

[a] Crops did not germinate or died at an early stage.

Table V. Recoveries of Aldrin and Dieldrin from Soils and from Crops Grown on Plots Treated with Aldrin 3 Years Previously

(Colorimetric analyses)

	Sandy Loam						*Miami Silt Loam*					
	Aldrin Applied to Soil in 1954, Lb./5 Inch Acre											
	2		20		200		2		20		200	
	Aldrin (A) and Dieldrin (D) Recovered, P.P.M., in 1957											
	A	*D*	*A*	*D*	*A*	*D*	*A*	*D*	*A*	*D*	*A*	*D*
	From soils											
Fall	0.00	0.09	0.08	1.01	5.66	15.2	0.00	0.17	0.14	0.84	12.3	12.86
	0.05[a]		0.59[a]		13.0[a]		0.03[a]		0.47[a]		15.65[a]	
	From crops											
Carrots	0.00	0.09	Traces	0.60	4.73	18.8	0.00	0.08	0.03[a]	0.80		13.6
	0.09[a]		0.64[a]		3.76[b]		0.07[a]		0.93[a]		9.86[b]	
Beets	[c]		[c]		[c]		0.00	0.00	0.00	0.06	[c]	
Cucumbers	0.00	0.22	0.00	0.36	[c]		0.00	0.09	0.00	0.14	[c]	
Potatoes	0.00	Traces	0.00	0.23	0.42	3.66	0.00	0.00	0.00	0.15	0.37	2.34
Radishes	[c]		0.64[a]		[c]		0.00	0.00	0.00	0.18	0.76	2.72
Rutabaga	0.00	Traces	0.00	0.31	[c]		0.00	0.06	0.00	0.10	0.18	1.88

[a] Bioassay-direct feeding method. [b] Bioassay on aldrin extract. [c] Plants did not germinate or died at an early stage.

available, to draw any definite conclusions.

Two years after soil treatment, carrots were grown on DDT-treated plots and analyzed for DDT content (Table III). Carrots grown on a sandy loam contained DDT which amounted to 10% of the quantity (in p.p.m.) found in the soil at the end of the growing season. Carrots did not contain any DDT, when grown in a Miami silt loam which had been treated at the rate of 10 pounds per acre. However, in plots (Miami silt loam), which had been treated with DDT at the rates of 100 and 1000 pounds per acre, 7.3 and 1.3% of the amount found in the soils (32.3 and 322 p.p.m.) at the end of the growing season, was recovered from the carrots, grown in these soils. In a muck soil, only those carrots which grew in a plot containing 775 p.p.m. of DDT, had absorbed the insecticide, although to a relatively small extent.

In 1957, three years after soil treatment, six different crops were grown in the DDT-treated plots (Table IV). More insecticide was absorbed from a sandy loam than from a Miami silt loam, and relatively more DDT had been absorbed by crops grown on those plots with the smallest amount of DDT. Potatoes, for example, grown in sandy loam plots, had the insecticide absorbed to an extent of 8.8 and 3.1% of the amounts (18.5 and 238 p.p.m., respectively) found in soils in which they had grown. In Miami silt loam only 3.8 and 1.3% of the amounts of DDT (24.0 and 302 p.p.m.) found in the soil, were recovered from the unpeeled tubers.

Insecticides which are more persistent in those soils having received the highest application rate (*11*), are probably less available for absorption, if they are more concentrated within the soil.

Regular applications of DDT to crops over a period of 4 to 10 years resulted in some contamination of the soil (*9*). However, the amount of DDT found in those crop soils did not exceed 2 to 3 p.p.m. Therefore, the quantity of this insecticide which might be absorbed into crops under usual agricultural conditions, is definitely not an alarming one.

The analytical results obtained from crops grown in aldrin-treated soils, are summarized in Tables III and V. When carrots and soils were analyzed for aldrin by chemical and bioassay methods, no aldrin was found chemically in some cases, yet mortalities were obtained when *Drosophila* flies were exposed to either carrots or soils. This seemed to indicate that another toxicant was present, which caused insect mortality. It proved to be dieldrin. In all cases, where toxicants were present within the crops investigated, more dieldrin than aldrin was found within those crops. Because the aldrin-treated soils, in which the various crops grew contained aldrin as well as dieldrin, it was impossible to determine whether the dieldrin found within the crop was absorbed from the soil or formed from the absorbed aldrin within the plant tissue.

Standard curves prepared with aldrin or dieldrin for the determination of LD_{50} values for *Drosophila* showed that aldrin was 1.8 times more toxic than dieldrin, when flies were used in a direct exposure method. Because all the soils and crops were measured against an aldrin standard only, the results obtained by bioassay (direct exposure) are actually higher than those presented in Tables III and V. In nearly all cases carrots contained more aldrin and dieldrin within their tissue than any other crop investigated. The amount of aldrin or dieldrin recovered from carrots was found to be very similar to the amount (in p.p.m.) present in the soil, in which the carrots were grown.

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