

## Insecticidal Residues in Various Crops Grown in Soils Treated with Abnormal Rates of Aldrin and Heptachlor

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A Carrington silt loam soil was treated with aldrin and heptachlor at rates of 5 and 25 pounds per 5-inch acre. The soil and crops grown on it were analyzed by colorimetric analyses, paper chromatography, and a bioassay procedure for residues of the applied aldrin and heptachlor, or their oxidation products: dieldrin and heptachlor epoxide. Data are presented on radishes, beets, potatoes, onions, carrots, cucumbers, lettuce, Lima beans, peas, and alfalfa. The potentiality of translocation from soils into crop tissues is demonstrated under these abnormal conditions of high insecticide dosages. However, it appears likely that treatment of soil with insecticide levels normally used for insect control would yield none or only trace amounts of insecticidal residues within the crop tissues investigated.

**D**URING RECENT YEARS various workers have considered insecticidal residues within crops which were grown in insecticide-contaminated soils (1, 3, 5, 15, 16). Recently (6) it was shown that under extreme experimental conditions several chlorinated hydrocarbon insecticides were translocated into pea vines from a quartz sand treated at 30 p.p.m.

To obtain more data under actual field conditions, loam plots were treated in 1958 and 1959 with aldrin and heptachlor. Various crops grown on these soils were analyzed for insecticidal residues.

### Procedure

**Soil Treatment and Sampling.** In May 1958, twelve 30 × 40 foot plots (Carrington silt loam) were staked out, leaving 20-foot buffer strips in between. Two insecticides, two application rates, and two replicates each were used for a total of eight experimental plots. Aldrin (A) or heptachlor (H) was applied at rates of 5 and 25 pounds per 5-inch acre. The four remaining ones were used as controls, although they were treated with solvent-emulsifier solutions which differed from the aldrin or heptachlor formulations only in that the insecticides were absent. Application rates of 5 and 25 pounds per 5-inch acre were chosen in order to have residue levels within the range of the sensitivity of the analytical methods. It was felt

that in such a way more accurate data might be obtained. In addition, it was intended to find out if higher soil residue levels (treatment with 25 pounds per acre) would result in relatively higher residues within the crops.

The insecticidal application involved a thorough mixing of a measured amount of an emulsifiable concentrate with 20 gallons of water. Each experimental plot was subdivided into five 6 × 40 foot strips in the south-north direction and five 8 × 30 foot strips in the east-west direction. Two-gallon quantities of the diluted emulsion were then spread as uniformly as possible with a sprinkling can over each of the 10 strips. This crosswise treatment was used to attain a more uniform application. Immediately after treatment all plots were rototilled to a depth of 4 to 5 inches.

Soils to which insecticides had been applied at 5 pounds per 5-inch acre in 1958 were retreated at the same rate in 1959. However, the treated areas in the second year of the experiment were reduced to 30 × 24 feet, because the remainder (30 × 16 feet) of each plot was in alfalfa which had been seeded one year previously. After retreatment of the four plots to which 5 pounds per 5-inch acre of aldrin or heptachlor had been applied in 1958, all 12 plots were rototilled to a depth of 4 to 5 inches.

Soil samples were collected in 1958

immediately after the insecticide application, as well as 2 and 4 months later. In 1959, however, samples were collected in the spring (after treatment) and fall only. The sampling area had a size of 30 × 24 feet which was not covered with alfalfa. Only the initial soil sample in 1958 was collected from the total area treated (30 × 40 feet).

Sampling was done with a soil auger, and 30 cores (3/4 inch in diameter, 6 inches long) were collected from each 30 × 24 foot area. Only the initial soil sample in 1958 consisted of 40 cores.

### Crop Growth and Crop Sampling.

In May 1958 and in May 1959 various crops were seeded in 30-foot rows of all treated and untreated plots. The crops grown during 1958 were radishes (Early Scarlet Globe), beets (Detroit Dark Red), potatoes (Russet Sebago), onions (Yellow Globe Danvers), carrots (Red Cored Chantenay), cucumbers (Straight Eight), lettuce (Great Lakes), Lima beans (Fordhook 242), and alfalfa (Vernal) (Figure 1). In 1959 the same crops were grown, except that peas (Wilt Resistant Alaska) were grown instead of Lima beans.

To eliminate the sampling problem, all the edible parts of a particular crop grown in one 30-foot row were collected at harvest time and brought into the laboratory for processing. Each edible plant part was then brushed in warm water, to remove adhering soil particles. After that, the crop was rinsed with acetone by means of a wash bottle and immediately thereafter rinsed with warm water. The clean crop parts were macerated with a food grinder, the ground material was mixed, and aliquots were placed in plastic bags within 1-quart ice cream cartons, frozen, and held until extraction. Seeds of beans and peas were not washed, but ground, and then processed as described. Alfalfa grown on plots

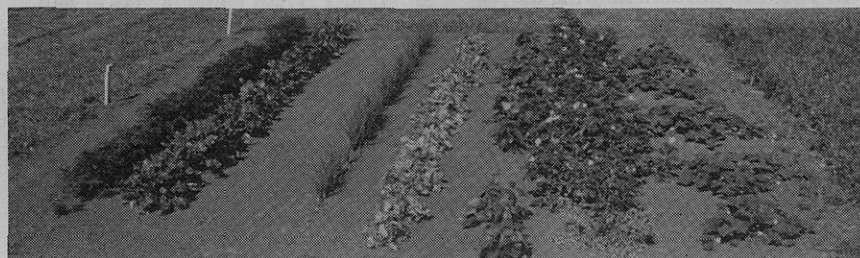


Figure 1. View of experimental plots in 1958

treated in May 1958 was sampled on June 9, 1959. The plants were 10 to 12 inches high and the harvest included all the plant parts more than 1 to 2 inches above the soil. From each plot between 2500 and 3500 grams of plant material was obtained by random sampling and harvesting by a procedure which ensured no surface contamination from the soil. After that the plants were passed through a food grinder and handled as described above.

Each potato tuber, after having been cleaned, was cut in two equal parts. One half was then peeled, resulting in peels and pulp. All the unpeeled portions, representing "whole potatoes," were ground, mixed, and subsampled for freezing and future analyses. The same was done with the peels as well as with the pulp, in order to find out what percentage of the total amount of residues being found within whole potatoes might be located in either the potato peels or the potato pulp.

#### Analytical Methods

**Colorimetric Analyses.** The handling of the soil samples, including extraction, clean-up, and analysis, was done as previously described (7-9).

When crops were analyzed, the frozen plant material was thawed and 300-gram aliquots were mixed with 600 to 700 grams of anhydrous sodium sulfate, and kept overnight in a refrigerator. Then the extraction of the crop material as well as the purification of the extracts was performed by a described procedure (6).

The extracts of the 1959 crops, grown in aldrin-treated soils, were separated by column chromatography into the aldrin and dieldrin (D) fractions. Each fraction was then subdivided into two equal aliquots. One aliquot was then treated with acetonitrile (4) to remove waxes. This portion was used for paper chromatography and bioassays, while the other was used for the colorimetric analyses as described by O'Donnell *et al.* (11, 12). Known amounts of aldrin added to crop material were recovered to an extent of 82 to 96% and known amounts of dieldrin to an extent of 85 to 98% as determined by colorimetric analysis.

Fractions of extracts containing heptachlor or heptachlor epoxide (HO) were analyzed by the Polen-Silverman method (13, 14). In this case, waxes were removed by an acetonitrile treatment (4) prior to analyses. Known amounts of heptachlor added to crop material were recovered to an extent of 84 to 98% and known amounts of heptachlor epoxide to an extent of 87 to 98% as determined by colorimetric analysis.

Whenever possible, analyses were run in duplicate, using a soil or a crop blank for the determination of appar-

**Table I. Recoveries of Aldrin (A), Dieldrin (D), Heptachlor (H), and Heptachlor Epoxide (HO) Residues**

[From a Carrington silt loam and crops grown in May 1958 on aldrin- and heptachlor-treated plots (colorimetric analyses)]

		Insecticides Applied to Soil, Lb./5-Inch Acre				
		Aldrin		Heptachlor		
		5	25	5	25	
Recovered from Soils, July 21, 1958, P.P.M.						
	A	1.21	9.77	H	2.17	10.78
	D	0.36	1.31	HO	0.21	0.63
	T <sup>a</sup>	1.57	11.08	T	2.38	11.41
Recovered from Crops at Harvest, P.P.M.						
Radishes	A	0.03	0.28	H	0.00	0.14
	D	0.06	0.24	HO	0.13	0.45
	T	0.09	0.52	T	0.13	0.59
Beets	A	0.00	0.07	H	0.00	0.13
	D	0.07	0.18	HO	0.08	0.29
	T	0.07	0.25	T	0.08	0.42
Potatoes	A	0.05	0.53	H	Traces	0.66
	D	0.09	0.67	HO	0.14	0.56
	T	0.14	1.20	T	0.14	1.22
Onions	A	0.00	Traces	H	Traces <sup>b</sup>	Traces <sup>b</sup>
	D	0.00	0.05	HO	Traces <sup>b</sup>	0.02 <sup>b</sup>
	T	0.00	0.05	T	Traces	0.02
Carrots	A	0.15	0.94	H	0.47	3.55
	D	0.09	0.32	HO	0.08 <sup>b</sup>	0.43 <sup>b</sup>
	T	0.24	1.26	T	0.55	3.98
Cucumbers	A	0.00	0.00	H	0.02	0.04
	D	0.07	0.07	HO	0.08	0.11
	T	0.07	0.07	T	0.10	0.15
Lettuce	A	0.04	0.15	H	0.05 <sup>b</sup>	0.27
	D	0.12	0.26	HO	0.05 <sup>b</sup>	0.29
	T	0.16	0.41	T	0.10	0.56
Beans (seeds)	A	0.00	0.00	H	0.00	0.00
	D	0.00	0.00	HO	0.00	0.00
	T	0.00	0.00	T	0.00	0.00

<sup>a</sup> Sum of aldrin and dieldrin or heptachlor and heptachlorepoide, respectively.

<sup>b</sup> Bioassay.

ent insecticide content. The analytical procedure for each analysis was checked by adding known amounts of insecticide to insecticide-free samples. The unknowns were then calculated on the basis of the values obtained from the known amounts after making suitable correction for apparent insecticide content. Results were expressed in parts per million based on the dry weight for soils and the fresh weight for plants.

In many cases colorimetric absorption curves were established for the recovered residue. In this way it could be checked if the absorption peak obtained from the unknown sample coincided with the peak obtained from a sample to which a known amount of an insecticide had been added.

In addition to the colorimetric analyses, fractions of extracts containing aldrin, dieldrin, heptachlor, or heptachlor epoxide were used for ascending paper chromatography as described by Mitchell (10).  $R_f$  values for the unknowns were established and compared to  $R_f$  values obtained from reference grade insecticides. Data were reported as zero residue when two or all of the methods used (colorimetric readings, absorption curves, paper chromatography) indicated the absence of insecticidal residues. When the results obtained by only one of the methods

were negative, the data were reported as trace residues.

**Bioassay.** It was felt that an analysis of the insecticidal residues by a biological method which assays the toxicity of a chemical would be extremely important. Therefore, some of the crops grown in 1958 and all of the crops grown in 1959 were bioassayed. *Drosophila melanogaster* Meig. was used as the test insect (2). The flies were exposed to residues obtained from aliquots of the same extracts used for the colorimetric analyses. Usually three different amounts of the same extract were tested by pipetting a given amount into each of two jars ( $2\frac{3}{4}$  inches in diameter and 3 inches deep) containing 0.5 ml. of a corn oil solution (1 ml. of corn oil in 200 ml. of hexane). The solvent was then evaporated by placing the jars at the opening of a fume hood. After a fine screen had been inserted into the screw top, 50 flies were introduced through a hole in the screen of each of the six jars (three different amounts of extracts in duplicate). Mortality counts were made 21 to 24 hours after exposure of the flies. Mortalities obtained with unknown samples were compared to dosage-mortality curves obtained from serial dilutions of reference grade insecticide. Results were reported as traces when mortalities obtained were below 15%.

The bioassay method was five to 10 times more sensitive than the available chemical methods. Therefore, the bioassay was the more dependable analysis when low residue levels were present.

## Results and Discussion

Data obtained from analyses of soils and crops are presented in Tables I, II, and III, where the figures represent

averages of analytical results obtained from soils and from crops grown on two replicated plots. The standard deviation obtained for data (Tables I, II, III) was:

**Table II. Recoveries of Aldrin (A), Dieldrin (D), Heptachlor (H), and Heptachlor Epoxide (HO) Residues**

(From a Carrington silt loam and crops grown in 1959 on aldrin- and heptachlor-treated plots)

		Insecticides Applied to Soil, Lb./5-Inch Acre							
		Aldrin				Heptachlor			
		5 <sup>a</sup>		25 <sup>b</sup>		5 <sup>a</sup>		25 <sup>b</sup>	
		Recovered from Soils, <sup>c</sup> P.P.M.							
	A	1.78		3.10		H	2.00		4.22
	D	0.66		1.83		HO	0.39		0.78
	T <sup>d</sup>	2.44		4.93		T	2.39		5.00
		Recovered from Crops at Harvest, P.P.M.							
		Chemical anal.	Bio-assay	Chemical anal.	Bio-assay	Chemical anal.	Bio-assay	Chemical anal.	Bio-assay
Radishes	A	Traces	0.03	0.05	0.05	H	Traces	0.04	Traces
	D	0.06	0.07	0.13	0.15	HO	0.12	0.12	0.12
	T	0.06	0.10	0.18	0.20	T	0.12	0.16	0.12
Beets	A	Traces	0.01	Traces	0.01	H	0.03	0.05	0.03
	D	0.09	0.06	0.17	0.14	HO	0.10	0.10	0.12
	T	0.09	0.07	0.17	0.15	T	0.13	0.15	0.15
Potatoes (whole)	A	0.05	0.03	0.11	0.11	H	0.09	0.14	0.29
	D	0.15	0.18	0.31	0.30	HO	0.26	0.31	0.49
	T	0.20	0.21	0.42	0.41	T	0.35	0.45	0.78
Potatoes (peels)	A	0.27	0.26	0.63	0.65	H	0.93	0.96	3.03
	D	0.91	0.91	1.82	2.10	HO	1.20	1.52	2.35
	T	1.18	1.17	2.45	2.75	T	2.13	2.48	5.38
Potatoes (pulp)	A	Traces	Traces	Traces	Traces	H	0.00	0.00	Traces
	D	0.06	0.06	0.16	0.14	HO	0.13	0.14	0.24
	T	0.06	0.06	0.16	0.14	T	0.13	0.14	0.24
Onions	A	Traces	0.00	Traces	0.00	H	*	0.00	*
	D	0.03	0.02	0.05	0.05	HO	*	0.03	*
	T	0.03	0.02	0.05	0.05	T	*	0.03	*
Carrots	A	0.25	0.30	0.36	0.43	H	0.97	1.26	1.34
	D	0.23	0.23	0.55	0.59	HO	*	0.25	*
	T	0.48	0.53	0.91	1.02	T	*	1.51	*
Cucumbers	A	Traces	Traces	Traces	0.01	H	0.03	0.04	0.04
	D	0.17	0.14	0.17	0.16	HO	0.10	0.12	0.13
	T	0.17	0.14	0.17	0.17	T	0.13	0.16	0.17
Lettuce	A	Traces	Traces	0.03	0.02	H	Traces	0.03	Traces
	D	0.07	0.06	0.17	0.18	HO	0.03	0.03	0.05
	T	0.07	0.06	0.20	0.20	T	0.03	0.06	0.05

Peas (seeds and pods) (6).

<sup>a</sup> Treated at 5 lb./5-inch acre in May 1958 and May 1959.

<sup>b</sup> Treated 25 lb./5-inch acre in May 1958 only.

<sup>c</sup> Averages of spring and fall analyses as obtained by chemical analyses.

<sup>d</sup> Sum of aldrin and dieldrin or heptachlor and heptachlor epoxide, respectively.

<sup>e</sup> Analytical data not available.

**Table III. Recoveries of Aldrin (A), Dieldrin (D), Heptachlor (H), and Heptachlor Epoxide (HO)**

(From a Carrington silt loam and from alfalfa grown in 1959 on plots treated with aldrin or heptachlor one year previously (colorimetric analyses))

		Insecticides Applied to Soil in May 1958, Lb./5-Inch Acre			
		Aldrin		Heptachlor	
		5	25	5	25
		Recovered from Soils, <sup>a</sup> P.P.M.			
	A	0.22	4.04	H	0.60
	D	0.98	4.18	HO	0.74
	T <sup>b</sup>	1.20	8.22	T	1.34
		Recovered from alfalfa leaves and stems, <sup>c</sup> P.P.M.			
	A	0.00	0.00	H	Traces
	D	0.06	0.08	HO	0.10
	T	0.06	0.08	T	0.10

<sup>a</sup> Fall sample.

<sup>b</sup> Sum of aldrin and dieldrin or heptachlor and heptachlor epoxide, respectively.

<sup>c</sup> Bioassay results for alfalfa grown on aldrin-treated soils not available. Bioassays on alfalfa grown on heptachlor-treated soils resulted in a total of 0.13 p.p.m. (5 lb./acre treatment) and 0.12 p.p.m. (25 lb./acre treatment), respectively.

Soils on which vegetables were grown and which had been treated in two consecutive years at a rate of 5 pounds per 5-inch acre showed a slight increase in the total amounts of residues recovered in the fall of 1959 (aldrin + dieldrin = 1.88 p.p.m., heptachlor + heptachlor epoxide = 2.20 p.p.m.) as compared to the amounts found within the soils in the fall of 1958 (1.28 and 1.88 p.p.m., respectively). However, in the fall of 1959 soils which had been treated 17 months previously at 25 pounds per 5-inch acre contained about half the amount of residues obtained in the previous year (fall 1958). The amount of epoxides in per cent of the total residues recovered from each treated plot was larger in 1959 than in 1958. Although aldrin is more readily converted in soils to dieldrin than is heptachlor to its epoxide, the total amounts of residues recovered from aldrin- and heptachlor-treated soils were very similar, because in soils aldrin is somewhat less persistent than heptachlor (9).

However, residue data obtained from crop analyses seem to indicate that the rate of epoxidation of aldrin and heptachlor within the plant tissue was in many cases very similar. Moreover, it is dependent on the particular crop. This could be due to the presence of certain biological systems which might effect the epoxidation of aldrin and heptachlor or to different absorption rates for the parent compounds and their epoxides from the soils.

Of all crops tested, carrots were outstanding, in that they contained the largest amounts of insecticidal residues. Those grown in plots treated at 5 pounds per 5-inch acre in 1958 and 1959 contained more insecticidal residues in the second than in the first year of the experiment. However, the opposite was true of carrots grown in soils treated at 25 pounds per 5-inch acre in 1958 only.

The proportion of dieldrin found in carrots ranged from 25 to 60% of the total residue (aldrin and dieldrin) recovered from the plant tissue. The respective figures for heptachlor epoxide were 11 to 21%.

Unpeeled potatoes contained more

insecticidal residues than any other crop, with the exception of carrots. Slightly more toxicants were recovered in 1959 than in 1958 in unpeeled potatoes grown in the plots treated with 5 pounds per acre and considerably less were recovered in 1959 than in 1958 from potatoes grown in the 25 pounds per acre treated plots. The amounts of dieldrin in unpeeled potatoes ranged from 56 to 75% of the total residue recovered from the whole potatoes. The respective figures for heptachlor epoxide ranged from 46 to 100%.

When potato peels and pulp were analyzed separately, it was found that 70 to 75% of the total residue in potatoes grown in aldrin-treated soil was located within the peels. Potatoes grown in heptachlor-treated soils contained 71 to 78% of their total residues within the peels. The amounts of toxicants recovered from potato pulp were similar to those found in radishes and beets. In 1959 these latter two crops contained residues which ranged from 0.06 to 0.13 p.p.m. in plants obtained from the 5 pounds per acre treated soils and from 0.12 to 0.18 p.p.m. in plants obtained from the 25 pounds per acre treated soils.

Onions were unique among the root crops because they either did not contain any insecticidal residues or the amounts found were below 0.05 p.p.m.

Among the nonroot crops tested, lettuce contained the highest amounts of insecticidal residues, which ranged from 0.10 to 0.56 p.p.m. in 1958 and from 0.03 to 0.20 p.p.m. in 1959.

The major part of the toxicants recovered from lettuce, cucumbers, or alfalfa was in the form of dieldrin or heptachlor epoxide.

Cucumbers and alfalfa were the only plants in which the absolute amounts of residues recovered from either crop were similar on both the 5 and the 25 pounds per acre insecticide-treated soils. Therefore, the highest amounts of toxicants in the plant relative to the soil were found in those plants which had been grown in the less contaminated soils.

Seeds of Lima beans or peas (6) did not contain any residues.

From the data presented it is obvious that insecticidal residues were found within the edible parts of crops grown in insecticide-treated loam soils. The amounts recovered varied for each crop investigated. In most cases the absolute amounts of residues recovered from crop tissues were higher in those plants which were grown in the most contaminated soils.

A more valuable figure for the understanding of the problem of insecticidal translocation from soils into crops might be the relative amount of toxicants recovered. This is the total amount of toxicants recovered from crop tissues in percent of the total amounts present in

**Table IV. Probable Insecticidal Residues (Aldrin Plus Dieldrin or Heptachlor Plus Heptachlor Epoxide)**

[Crops grown on a Carrington silt loam containing 2 pounds/6-inch acre (1 p.p.m.) of toxicants]

	Crop Residue, % of Soil Residues <sup>a</sup>		Probable Crop Residues, <sup>b</sup> P.P.M.	
	A <sup>c</sup>	H <sup>d</sup>	A <sup>c</sup>	H <sup>d</sup>
Carrots	15.9 ± 3.42	31.7 ± 6.18	0.16	0.32
Potatoes (unpeeled)	8.9 ± 0.74	11.8 ± 3.75	0.09	0.12
Potatoes (pulp)	2.8 ± 0.40	5.1 ± 0.40	0.03	0.05
Radishes	4.2 ± 1.23	4.6 ± 1.33	0.04	0.04
Beets	3.5 ± 0.79	3.9 ± 0.93	0.04	0.04
Onions	<1.0	<1.0	<0.01	<0.01
Lettuce	5.2 ± 2.82	2.9 ± 1.77	0.05	0.03
Cucumbers	3.8 ± 2.27	3.6 ± 1.46	0.04	0.04
Peas (seeds)	0.0	0.0	0.00	0.00
Beans (seeds)	0.0	0.0	0.00	0.00
Alfalfa	3.0 ± 2.00	4.4 ± 3.10	0.03	0.04

<sup>a</sup> Based on field experiments conducted during 1958 and 1959.

<sup>b</sup> Calculated from experimental data as presented in left column of this table, assuming that the proportion of insecticide translocating from a soil into the crops is independent of the concentration of the insecticide within the soil.

<sup>c</sup> Grown in aldrin-treated soils.

<sup>d</sup> Grown in heptachlor-treated soils.

the soil (average of spring and fall soil samples) during the growing season.

These relative figures obtained from one plant variety were in most cases similar on both the 5- and 25-pounds per acre treated plots and during both of the two years of the experiment. Therefore, it might be possible to approximate the amounts of insecticidal residues in a particular crop, once the amount of aldrin or heptachlor residues in the soil during the growing season as well as the crop residue in per cent of the soil residue was known. Such an attempt has been made, assuming that a soil treated at a rate of 2 pounds per 6-inch acre has an insecticide concentration of 1 p.p.m. Under more normal conditions the total amount of residues in all crops, with the exception of carrots and unpeeled potatoes, would range from 0.00 to 0.05 p.p.m. (Table IV). These theoretical recoveries are maximum figures, since a 2 pounds per 6-inch acre treatment at the beginning of the growing season will result in much smaller soil residues with time. In the case of radishes, which grew during the first month after soil treatment, the insecticidal residues in the soil were larger than toward the end of the growing season. Therefore the relative amounts of insecticides found in radishes would be somewhat smaller than presented in Table IV. In the case of potatoes, which were harvested in September, the relative amounts of residues within the tubers might be slightly higher than presented in Table IV. Field studies similar to those described in this paper are under way, using normal dosages of aldrin and heptachlor.

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