Dieldrin and Heptachlor Epoxide Residues in Fat from Hogs Foraging on Corn Stover in Insecticidally Treated Fields[1]

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The use of swine to glean corn fields after picking is a well-established practice in the Middle West. Soil which has been treated with chlorinated hydrocarbon insecticides in the spring for control of soil insects contains appreciable residues in the fall. Animals feeding on the stover or rooting in treated soil are exposed to pesticide contamination as a result of this activity. Davich et al. (1), demonstrated that swine foraging in a pasture treated with aldrin, dieldrin, or heptachlor would accumulate dieldrin or heptachlor epoxide in adipose tissues. The purpose of the present study was to determine whether or not residues of chlorinated hydrocarbon insecticides would accumulate in adipose tissues of swine permitted to forage on stover grown in soils treated for corn root worm control.

Methods and Procedures:

During the three year period of the study, the same three corn fields were utilized. (Fig. 1) One field was treated with heptachlor, one with aldrin, and the third was kept as an untreated check. The latter field has never been treated with insecticides but was sufficiently close (approximately 20' of fence row and farm road away) to the other fields so that spray drift or dust might contaminate it to some slight degree.

Experiment 1. 1967-68

Of the three fields utilized in this experiment, Field 1 (Fig. 1) had been treated with 3 lbs. actual heptachlor per acre in May 1965 and was retreated at the same rate in May 1967 (Table 1). Field 2, adjacent to and north of Field 1 was sprayed with 3 lbs. of actual aldrin per acre in May of 1966 and again in May 1967. The check field described above was labelled field 3.

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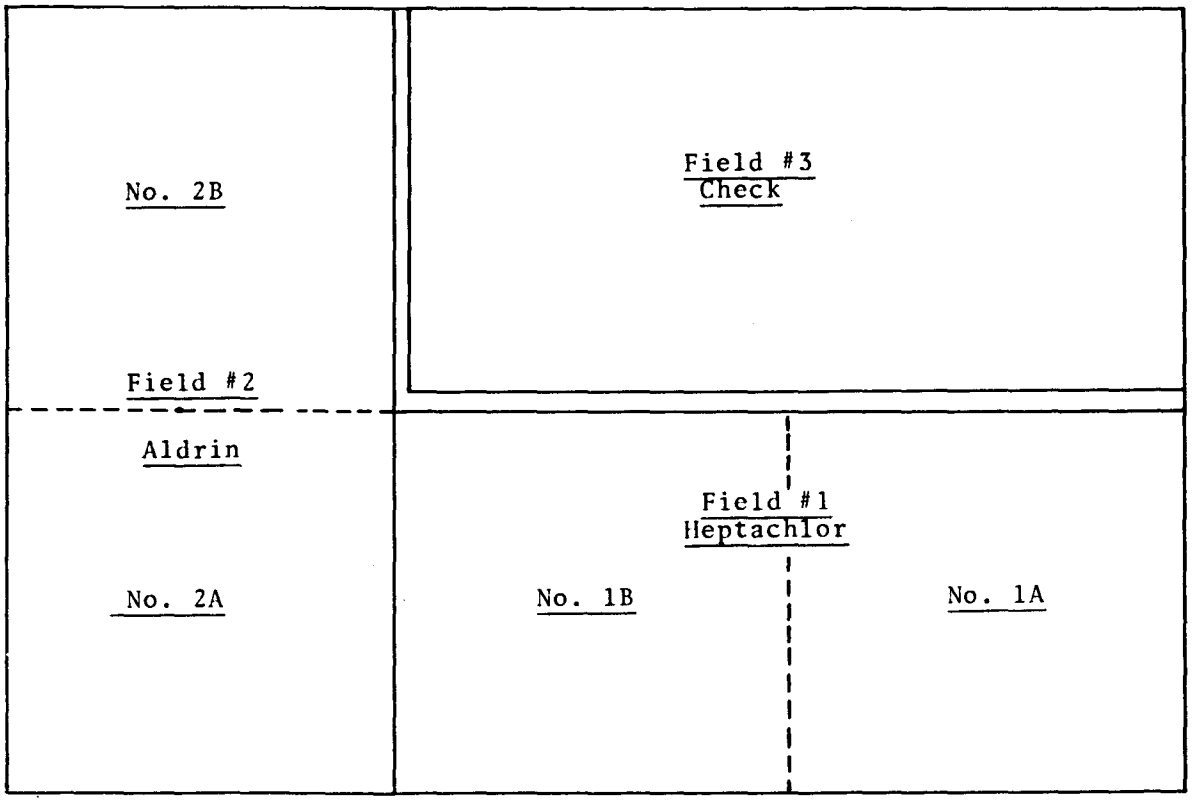

Fig. 1. Layout of Experimental Fields

– – – – – = Denotes Fences Utilized in 1969-70 Trials

Table No. 1 Chronology of Field Treatments

Date	Field #1 1A	Field #1 1B	Field # 2 2A	Field # 2 2B	Field #3
May 1965	3#Hepta.1/	3#Hepta.	None	None	None
May 1966	None	None	3#Aldrin2/	3#Aldrin	None
May 1967	3#Hepta.	3#Hepta.	3#Aldrin	3#Aldrin	None
May 1968	None	None	None	None	None
May 1969	3#Hepta.	None	3#Aldrin	None	None

1/ 3# Hepta. = 3# Actual Heptachlor per Acre.

2/ 3# Aldrin = 3# Actual Aldrin per Acre.

Table 2

Residues of soils treated with Aldrin and Heptachlor

Date Sampled	Aldrin Treated		Heptachlor Treated		Control
	Aldrin	Dieldrin	Heptachlor	Hepta. Epoxide	
June 16, 1967	0.740	0.310	0.270	0.240	0.002
Nov. 27, 1968	.021	.171	.012	.058	.002
May 26, 1969	.030	.243	.025	.094	.002
Prespray March 28, 1970					
Field 1A			.295	.157	
Field 1B			.022	.140	
Field 2A	.166	.373			
Field 2B	.063	.259			
June 2, 1970					
Field 1A			.097	.093	
Field 1B			.021	.135	
Field 2A	.048	.400			
Field 2B	.081	.232			

On March 6, 1967, 32 feeder pigs weighing an average of 65 lbs. were purchased. These animals were raised by a single grower and had been on concrete since birth. Two animals were sacrificed immediately and fat samples taken for reference analysis. The remaining pigs were randomly divided into 3 groups and one group was placed in each of the three fields. In addition to the stover in the fields, a mixture of ground corn and protein supplement was supplied the animals ad libitum.

The animals were confined to the test fields until May 10 (65 days) after which they were moved to a feed lot. The 1967 spray applications were made following removal of these hogs. At this time, all animals received only the ground corn-protein supplement mixture.

Nine animals (1 from each lot died of extraneous causes) from each group were slaughtered on June 21, 42 days after being placed in the feed lot. Average weight of the hogs at this time was 230 lbs.

Experiment 2. 1968-69

The same three fields utilized in 1967-68 were used during the course of this experiment. No further insecticides were applied to the area after the May 1967 applications. Corn was planted and harvested on these fields during 1968.

On December 17, 1968, 29 pigs averaging 40 lbs. each were purchased from the Herbert Davis-Purdue Agricultural Center. These pigs were from an insecticide-free area and were raised on concrete. Two pigs were sacrificed and body fat samples taken for reference analysis. The remaining 27 were divided into 3 lots of 9 each and allotted to the three fields. These animals foraged over the fields with no supplemental feed until February 25, 1969 (71 days). At this time, one pig from each of the areas was sacrificed and kidney and belly fat samples taken. At the same time, a complete ration of ground corn plus protein supplement was supplied the animals. They remained on the experimental ground throughout the experiment.

On April 14, 1968, the animals remaining on trial, (8 per field) were slaughtered and kidney and belly fat samples taken. The animals weighed an average of 215 lbs. and had been on the experimental fields a total 119 days.

Experiment 3. 1969-70

The same fields used in '67-'68 and '68-'69 were utilized in this experiment. In this case, however,

each of the two fields treated with insecticides in '65 and '67 was divided in half. One half was left untreated while the other was resprayed with its candidate insecticide on May 26 of 1969 at the same rate as before. (Table 1)

On December 3, 100 pigs were randomly assigned to the experimental fields at the rate of 20 per lot. Two animals were sacrificed from field 3 on December 5 for residue samples.

Nine pigs from each lot were taken on March 10 to a feed lot and 2 animals from each lot were sacrificed for sampling at this time. The remaining animals were continued in their respective fields. The animals which were moved to the feed lot were fed a corn-protein supplement diet until the conclusion of the experiment. Animals left in the fields were also supplied the same ration.

On March 31, the animals left in the treatment fields were slaughtered and fat samples taken at that time. They had been on the fields for 118 days.

The animals which had been moved to the feed lot were slaughtered on April 3 and fat samples were taken at that time. These animals had been exposed to the treatment fields for 98 days and were on clean feed for an additional 24 days for a total of 122 days on trial.

All samples of fat taken from the hogs were collected from the belly, the fat beneath the surface on the underside of the hog, and from plate fat, that fat lying adjacent to the kidneys. These samples were quick frozen and held at -15°C. until analyzed.

Results: Analysis techniques.

Green corn, corn ears, stover residues.

Residue analyses were made on green corn, corn ears, and corn stover in 1967 from both the aldrin and heptachlor treated plots. Green corn contained less than 0.002 ppm of heptachlor or its epoxide. The corn from the aldrin treated plot contained no appreciable aldrin but did contain a dieldrin residue of approximately 0.004 ppm. This residue approximates the minimum sensitivity of the analytical procedure employed. The corn ears collected from these plots contained no detectable residues of heptachlor, heptachlor epoxide, aldrin, or dieldrin. Corn stover from these fields was collected on November 29, 1967 and analyzed. Material from the heptachlor treated field contained 0.032 ppm of heptachlor epoxide. Material from the aldrin treated field contained 0.026 ppm of dieldrin. Since most of the

Table 3. Residues of Dieldrin and Heptachlor Epoxide in Soluble Lipids from Swine

Treatment Period		Hepta Epoxide		Dieldrin	
		Belly	Plate	Belly	Plate
1967-68					
Pretreatment	2[6/]	.014	---	.016	---
Slaughter(107 days)[1/]					
Treatment animals	9	.046 ± .017	.051 ± .023	.092 ± .018	.096 ± .015
Control animals	9	.004 ± .0007	.004 ± .0006	.009 ± .0027	.009 ± .0017
1968-69					
Pretreatment	2	.015	.014	.026	.038
Pre-ground feed(71 days)[2/]					
Treatment animals	1	.071	.071	.083	.080
Control animals	1	.010	.005	.014	.019
Slaughter(119 days)[3/]					
Treatment animals	9	.060 ± .042	.060 ± .043	.141 ± .066	.139 ± .064
Control animals	9	.003 ± .0005	.003 ± .0008	.004 ± .0009	.004 ± .0009
1969-70					
Pretreatment	2	.01	---	.01	---
Pre-ground feed(98 days)					
Field 1A	2	.032			
Field 1B	2	.034			
Field 2A	2			.122	
Field 2B	2			.036	
Field 3	2	.01		.01	
Slaughter(118 days)[4/]					
Field 1A	7	.044 ± .023			
Field 1B	7	.027 ± .008			
Field 2A	7			.054 ± .023	
Field 2B	8			.058 ± .029	

Table 3. (continued)

		Hepta Epoxide		Dieldrin	
		Belly	Plate	Belly	Plate
Slaughter (122 days)[5/]					
Field 1A	7	.038 ± .013			
Field 1B	4	.024 ± .001			
Field 2A	8			.034 ± .013	
Field 2B	6			.038 ± .028	
Field 3	8	.010		.010	

1/ 65 days on treated area followed by 42 days on concrete feed lot.

2/ Animals fed upon stover only for 71 days.

3/ Animals on treated area throughout experiment. Fed stover only for 71 days and supplemented with ground corn, protein for 48 more days.

4/ Animals on treated area throughout experiment. Fed stover only 98 days and supplemented with ground corn, protein for 20 more days.

5/ Animals fed stover untreated area only 98 days. After moving to concrete feed lot fed ground corn protein for 24 days.

6/ Number of animals analyzed.

stover was lying on the ground, it is speculated that the residues found were as a result of contamination with soil rather than through translocation of the residues from the soil to the plants.

The ground corn-protein supplement feed was sampled throughout the feeding periods and no heptachlor or aldrin residues were found in the feed samples. Traces approximating 0.001 ppm of heptachlor epoxide and 0.002 ppm of dieldrin were detected. These levels approached the minimum detectable residues of the methods employed.

Soil residues

Soil samples were taken throughout the three year period and residue studies made. Table 2 shows the results of analysis of the soil each year. The June 1967 soil samples were taken approximately one month after treatment; November 1968 samples were taken before the pigs were placed on the fields; and the May 1969 samples were taken immediately before the sprays were applied that year.

It becomes apparent that aldrin is quite rapidly broken down in the soil and it also is apparent that dieldrin replaces it as the aldrin breaks down. Dieldrin is very obviously much slower to break down in the soil than the aldrin.

Heptachlor is also very readily broken down and heptachlor epoxide builds up. However, unlike the dieldrin residues, heptachlor epoxide breaks down quite rapidly in the soil after a period of weathering.

Residues in Soluble Lipids

As may be determined in Table 3, pretreatment residues in the animals in all three years were at a very low level ranging from 0.038 ppm dieldrin in 1968-69 to less than 0.01 in 1969-70 with heptachlor epoxide levels at about 0.01 each of the three years. It is also apparent that in 1967-68 and again in 1968-69 but not in 1969-70, that very small amounts of dieldrin and heptachlor epoxide were found in animals on the control or untreated fields. This is probably due to the very small amounts of each material found in the ground corn-protein supplement feed supplied to these animals.

As may be seen in Table 3, measurable amounts of both heptachlor epoxide and dieldrin were detected in the animals whether they were confined to the treated areas throughout the treatment period or whether they were kept for varying periods on a non-contaminated surface i.e. concrete feed lot, after exposure to the contaminated areas.

From the data presented, it may be seen that very little loss of heptachlor epoxide occurred whether the animals were left on the treated areas for the full period or whether they were removed. The dieldrin-residue hogs showed definite losses in levels of residues when they were removed from the treated soils as may be noted in the different levels obtained in the 1969-70 Field #3 experiment.

Dobson et al. (2), have shown that dieldrin residues in hogs are naturally diluted during a fattening period following acquisition of the material. This is at least partially borne out in the data presented above.

Analysis of Residues:

Soil samples were collected from the experimental area on June 16, 1967; November 27, 1968; May 26, 1969; and March 28, 1970. Four replicates of 15 cores (3/4" X 6") were taken from each field in 1967. Ten cores per replicate were taken in 1968, 1969, and 1970. Residues were extracted from the soil with a 2:1 solution of hexane and isopropyl alcohol. The isopropyl alcohol was removed by water washes. The remaining hexane portion was put on a Florsil (used as received) column. An eluate of hexane was used to remove aldrin and heptachlor. An eluate of 15% ether in hexane was used to remove heptachlor epoxide and dieldrin from the 1967 soil samples. However, in 1968 and 1969 the eluate (from the column) containing heptachlor epoxide and dieldrin was adjusted to suitable volume and quantitated with an electron affinity detector with GLC as with adipose tissue.

Analysis of Adipose Tissues:

Samples of 25 gm of tissue were placed in a Laudres Homogenizer with 50 grams of sodium sulfate and 200 ml of hexane (distilled in glass) and blended for 3 minutes. The residue solution was filtered and the solvent removed by vacuum evaporation. Residue analyses are based on extractable lipids. A five gram portion of the extractable lipid was dissolved in 25 ml. hexane and extracted 4 times with 25 ml acetonitrile (hexane saturated). The combined acetonitrile extracts were diluted with 600 ml water and extracted twice with 100 ml hexane. The hexane extract was dried over sodium sulfate, filtered and the volume adjusted to 10 ml using a rotary evaporator. A 2.5 X 10 cm florsil column was washed with 50 ml hexane, the sample added to the column and eluted with 100 ml hexane to recover aldrin and heptachlor residues. The column was then eluated with 50 ml of 3% ether in hexane and discarded. The column was then eluted with 75 ml

of 6% ether in hexane to recover the heptachlor epoxide and dieldrin residues. The 6% ether fraction was reduced to approximately 5 ml on a rotary evaporator. An 11 mm X 4 cm Alumina A-540 column was washed with 20 ml hexane and the 6% ether fraction from the previous column was added. The column was eluted with 50 ml hexane followed by 5 ml benzene and discarded. The column was then eluted with 20 ml benzene to recover the heptachlor epoxide and dieldrin. The sample was evaporated to dryness and brought up to 10 ml volume in hexane. Final determination was by electron capture gas--liquid chromatography with a 5 ft. glass column containing 3% QF-1 and 2% SE-30 on Chromosorb W at 185°C. The minimum detectibility of heptachlor epoxide and dieldrin was .002 ppm.

Additional identification of heptachlor epoxide and dieldrin was achieved by partition coefficients (p-values) between hexane and acetronitrile. A pure dieldrin standard gave a value of .28 while 3 fat samples gave values of .30, .27, and .27. A pure heptachlor epoxide standard gave a value of .23 while 3 hog samples gave .24, .22, and .23. These figures are reasonably close to those of Beroza (3).

Summary:

Hogs pasturing on corn stover in insecticidally treated fields accumulated measurable residues of dieldrin and heptachlor epoxide. Hogs weighing between 40 and 60 pounds were placed on these treated fields for varying periods of time. Residues of heptachlor epoxide and dieldrin accumulated in the fat to levels of from 0.032 to 0.071 ppm. of heptachlor epoxide and from 0.080 to 0.141 ppm. of dieldrin. Removal of the animals to concrete slabs for further fattening for varying periods of time reduced but did not eliminate these residues.

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