Insecticide Residues in Wheat Grown in Soil Treated with Aldrin and Endrin

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Residues in spring wheat grown in soil treated with aldrin at 8 pounds per acre and endrin at 1/8, 1/2, 2, or 8 pounds per acre were determined by electron-capture gas chromatography. Forty-five (preboot stage) and 60 (25% headed) day-old plants and harvested straw grown in the two lower rates of endrin-treated soil contained no measurable amount (less than 2 p.p.b.) of endrin, whereas those grown in the 2- and 8-pound endrin-treated soil had 0.015 to 0.075 p.p.m. of endrin. Comparable samples from the aldrin-treated soil contained 0.014 to 0.025 p.p.m. of dieldrin. No residue was detected in the grain even when 8 pounds per acre of endrin or aldrin were incorporated into the soil.

In the past few years insecticides of the chlorinated hydrocarbon type have been shown to translocate from soils into various plants (*12*). Most of the quantitative work in this field has been directed toward vegetable crops (*6*, *7*, *9*, *10*). Recently Bruce, Decker, and Wilson (*1*) demonstrated the translocation of aldrin, dieldrin, heptachlor, and its epoxide into barley, corn, oats, soybeans, and peanut seeds grown on heptachlor- or aldrin-treated soil. They also noticed an apparent relationship between the oil content of the seed and the residues found therein. Corn and barley, having about 2 and 3% oil, respectively, grown in soil containing 1 p.p.m. of these pesticides had a barely detectable amount (2 p.p.b.) of residue; peanuts (ca. 50% oil content) grown in the same soil had 0.5 p.p.m. residue.

In the Canadian Prairie Provinces seed dressings of aldrin at the rate of 1/2 ounce per bushel of seed per acre for wireworm control and surface applications of endrin at 2 to 4 ounces per acre for cutworm control are recommended for cereal crops. Because of the persistent nature of these insecticides, residues could accumulate in the soil after a number of years' applications. The possibility of their translocation from soil into wheat grain under field conditions has never been investigated. However, Morley and Chiba (*13*) have recently demonstrated translocation of dieldrin in wheat grain (0.06 to 0.14 p.p.m.) and stems of plants grown in soil containing an abnormally high amount of dieldrin (20 p.p.m. or ca. 40 pounds per acre, 6 inches deep). The wheat was grown in the greenhouse in a mixture of vermiculite and sand and covered with aluminum foil to prevent volatilization of dieldrin. Because of the conditions of this experiment, it would be difficult to predict whether translocation of dieldrin or other chlorinated hydrocarbon pesticides could occur from soil into wheat grain under normal farming and insect control practices. Thus it was important to study this question.

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This paper reports on the translocation of aldrin (and dieldrin) or endrin into wheat plants grown in soil into which aldrin and different amounts of endrin had been incorporated prior to seeding to simulate varying levels of accumulated residues.

Method

Soil Treatments. Each soil treatment was replicated four times in a randomized plot (each 40 × 100 feet) complete block design. The soil was silt loam with 6% organic matter content. Endrin emulsion (2 pounds per Imperial gallon) was applied from a spray boom at 1/8, 1/2, 2, and 8 pounds and aldrin emulsion (2 pounds per Imperial gallon) at 8 pounds of toxicant per acre. The toxicants were applied to the soil surface in 16.5 Imperial gallons of liquid per acre, double-disked into the top 4 to 6 inches immediately after application, and packed to provide a firm seed bed. Seeding was done 8 days after treatment of the soil with a double-disk drill set to seed 2 1/2 inches deep. Each plot was subdivided into two 20 × 100 foot subplots. One subplot was seeded with spring wheat treated with 1/2 ounce of aldrin per bushel and the other subplot with untreated seed. The purpose of using treated seed on one half of each plot was to superimpose wireworm control on cutworm control, as farmers may use both in a given year.

Sampling of Soil, Wheat Plants, and Grain. A soil sample consisting of 10 cores (each 2 inches in diameter and 6 inches long from each of the subplots) was taken 8 and 138 days after spraying. The first set of samples was taken in May just before the plots were seeded and the second set of samples was taken in October after the grain was harvested. A representative sample of wheat plants (ca. 1 pound each) was collected from each of the subplots 45 (preboot stage) and 60 days (25% headed) after seeding. Similar samples of grain and straw were taken at harvest time (112 days after seeding). All the samples of soil, wheat plant, and grain were stored in plastic bags at 0° F. until they were analyzed. The plant and grain samples were washed with cold water before analysis.

Analytical Methods. All the samples were analyzed in duplicate by gas-liquid chromatography using a Model 600-D Aerograph Hi-Fi gas chromatograph with electron-capture detector and 5-foot × $^1/_8$-inch i.d. aluminum column packed with 4% SE30 on 80- to 100-mesh Chromosorb W and a carrier gas, oxygen-free nitrogen with a flow rate of 120 ml. per minute. The injector, column oven, and detector temperatures were 180°, 176°, and 175° C., respectively. The electrometer range and sensitivity were 1 and 4, respectively. Under these conditions the retention times of aldrin, dieldrin, and endrin were 3.2, 8.0, and 10.8 minutes. The amount of each pesticide present in a particular sample was determined by the internal standard method (*15*) using heptachlor epoxide (retention time 4.2 minutes) as the standard.

The method for extraction and cleanup of soil samples was that reported by Saha and Stewart (*16*). Air-dried soil samples (10 grams) were extracted with 1 to 1 hexane–2-propanol mixture, partitioned into petroleum ether, and chromatographed on a magnesia-Celite (1 to 1) column. The procedure for extraction and cleanup of wheat foliage and straw was the same as that described by Saha and Stewart (*16*) for rutabagas. These samples were extracted with acetonitrile, partitioned into petroleum ether, and chromatographed on a mixture of magnesia and Celite (4 to 1). The method for the analysis of wheat grain has been reported (*15*). Amounts of aldrin, dieldrin, and endrin added to soil were recovered to an extent of 95 to 100%. Recoveries of the same insecticides from fortified wheat plant materials were between 90 and 98% and those from wheat grain were almost quantitative (*15*). The minimum amount of these compounds detectable in the soil and plant materials was 2 p.p.b. and that in grain was 5 p.p.b.

Results and Discussion

Soil Residues. Since no soil sample was taken immediately after application of the toxicants, the actual amount applied to soil could not be determined. However, the amount of residues remaining 8 and 138 days after application can be used to estimate the disappearance of endrin and aldrin from soil and the conversion of aldrin into dieldrin in soil.

Assuming the weight of 1 acre of loam soil 6 inches deep to be 2,000,000 pounds, the amounts of toxicants recovered from soil were calculated (Tables I and II). Generally speaking, a higher proportion of residue remained in soil at the higher rates of applications—i.e., loss was more rapid from soil containing lesser amounts of endrin (Table I). Decker, Bruce, and Bigger (*3*) have shown that aldrin is rapidly lost by volatilization immediately after application. The low recovery of aldrin 8 days after application would appear to support this finding when only about 58% of the estimated applied dosage was recovered (Table II). Part of the aldrin present in the soil could have been lost by volatilization when the soil samples were being air-dried. The soil also could have received much less than the estimated amount of application in this particular instance. At the end of 138 days 28% of the residues was dieldrin, in agreement with the data obtained by Decker, Bruce, and Bigger (*3*) on the conversion of aldrin into dieldrin in soil.

Residues in Wheat Foliage and Grain. Wheat foliage samples from 2- and 8-pound endrin-treated plots contained between 0.015 and 0.075 p.p.m. of endrin, while no residue was detected at the two lower rates of application (Table I). Comparable samples from the aldrin-treated soil contained 0.014 to 0.025 p.p.m. of dieldrin (Table II). These amounts are rather low compared to the absorption of some chlorinated hydrocarbon pesticides by other plants. For example, King, Clark, and Hemken (*4*) found about 0.2 to 0.3 p.p.m. of heptachlor and its epoxide in alfalfa grown in soil treated with only 1 pound per acre of heptachlor. Straw samples had about the same or slightly more residue (fresh weight basis) than the green foliage (Tables I and II). However, straw had

Table I. Recoveries of Endrin Residue from Soil and from Wheat Plants and Grain Grown in Soil Treated with Endrin[a]

Endrin Applied, Pounds per Acre	Endrin, P.P.M.[b] Soil, Days after Application: 8	Soil, Days after Application: 138	Wheat Plants, Days after Seeding[c]: 45	Wheat Plants, Days after Seeding[c]: 60	Straw[c]
8	3.71 ± 0.05 92.8%[d]	2.3 ± 0.04 57.5%	0.045 ± 0.005	0.075 ± 0.007	0.050 ± 0.004
2	1.06 ± 0.06 106.0%	0.60 ± 0.03 60.0%	0.015 ± 0.006	0.017 ± 0.003	0.025 ± 0.003
8	0.09 ± 0.03 36.0%	0.03 ± 0.02 12.0%	[e]	[e]	[e]
2	0.02 ± 0.02 32.0%	0.004 ± 0.003 6.4%	[e]	[e]	[e]

[a] No residue detected in any wheat grain samples.
[b] Mean values for duplicate samples from each of eight subplots of four replicates with standard errors. Wheat plants and straw residues on fresh weight basis, soil residues on oven-dry weight basis. Moisture content of wheat plants and straw 75 and 6%, respectively.
[c] In addition to endrin residues wheat plant samples after 45 days and straw from aldrin seed treatment subplots had 0.003 p.p.m. dieldrin at all levels of soil treatment. No dieldrin residue detected in any other sample.
[d] Recovered, % of estimated applied dosage.
[e] No measurable residue (less than 2 p.p.b.).

Table II. Recoveries of Aldrin and Dieldrin Residues from Soil, Wheat Plants, and Grain Grown in Soil Treated with Aldrin at 8 Pounds per Acre[a]

	Residues, P.P.M.[b]				
	Soil, Days after Spraying		Wheat Plants, Days after Seeding		
	8	138	45	60	Straw
Aldrin	2.31 ± 0.07	1.30 ± 0.005	[c]	[c]	[c]
Dieldrin	[c]	0.50 ± 0.03	0.014 ± 0.004	0.025 ± 0.005	0.051 ± 0.004
Aldrin + dieldrin	2.31	1.80	0.014	0.025	0.051
	57.8%[d]	45.0%			

[a] No residue detected in grain.
[b] Mean values for duplicate samples from each of 8 subplots with standard errors. Wheat plants and straw residues on fresh weight basis, soil residues on oven-dry basis. Moisture content of wheat plants and straw 75 and 6%, respectively.
[c] Not measurable (less than 5 p.p.b. in grain and 2 p.p.b. in plant materials and soil).
[d] Recovered, % of estimated applied dosage.

only 6% water compared to 75% in the green plants.

No measurable amount (less than 5 p.p.b.) of endrin or dieldrin was detected in wheat grain grown in soil treated with even 8 pounds per acre of endrin or aldrin. In the light of the small amount of translocation of dieldrin into oats and barley grain observed by Bruce, Decker, and Wilson (*1*) and considering the low oil content (2%) of wheat grain (*14*), these results are not surprising. Apparently, the translocation of these pesticides is limited primarily to the leaves and stems of wheat plants. All the plant and grain samples were washed with cold water before they were analyzed. It is not very likely that the residues detected in the plant tissues resulted from contamination with soil.

Irrespective of the concentration of endrin in soil, the 45 day old plants and straw from the subplots, seeded with aldrin-treated seeds, had about 3 p.p.b. of dieldrin. The 60 day old plants, wheat grain, and soil after 138 days had no detectable amount of dieldrin. In the case of wheat grown from aldrin-treated seeds in aldrin-treated soil, similar amounts of dieldrin would be expected to be present in the 45 day old plants and straw samples as contribution from treated seeds. Since the standard errors in these cases were more than 3 p.p.b., it is not possible to separate the extent of translocation from soil and seed treatment. Burrage and Saha (*2*) obtained similar results in their experiments with wheat grown from aldrin-treated seeds.

Residues of chlorinated hydrocarbon pesticides are known to persist in soil long after their use. From a study of the accumulation and dissipation of residues resulting from the use of aldrin in soils, Decker, Bruce, and Bigger (*3*) have concluded that "under corn belt conditions in Illinois the probability that annual applications of aldrin over a period of 10 or more years will result in accumulation in excess of the annual application rate is remote." It is very unlikely that residues from recommended use of endrin and aldrin over an extended period will ever exceed the highest level of residues in soil used for the present study, even though the persistence of these insecticides in soil is influenced by soil types, rate of application, and temperature (*8*, *11*). Thus, under normal farming conditions, the use of aldrin and endrin for wireworm and cutworm control in wheat fields should not leave any detectable amount of these insecticides in the grain, although the straw and foliage may not be safe as cattle feed.

Korte and Arent (*5*) have recently shown that dieldrin is metabolized by rats to give at least six products, of which the major component is *trans*-6,7-dihydroxydihydroaldrin (86%). Possibly, plants also might metabolize dieldrin and endrin. A study of the metabolism of dieldrin and endrin in plants is under way, and the results will be reported later.

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