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Organochlorine Insecticide Residues in Quail, Rabbits, and Deer from Selected Alabama Soybean Fields

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Samples of edible meat were collected from bobwhite quail (*Colinus virginianus*), swamp and cottontail rabbits (*Sylvilagus aquaticus* and *S. floridanus*), and white-tailed deer (*Odocoileus virginianus*) found in or adjacent to selected soybean fields in Alabama and from areas with little or no history of insecticide use. Samples were analyzed for residues of organochlorine and organophosphate insecticides. DDT and its metabolites (DDT + DDE + DDD) were the only insecticides occurring consistently in the samples tested. Toxaphene, heptachlor epoxide, and dieldrin were detected in a small percentage of the animals. Total DDT residues (DDT + DDE + DDD reported on a lipid basis) averaged 17.08±, 3.00±, and 2.47± ppm, respectively, for bobwhite quail, white-tailed deer, and rabbits collected from treated soybean fields. Total DDT residues averaged 1.68±, 0.10±, and 0.05± ppm, respectively, for bobwhite quail, white-tailed deer, and rabbits collected from areas with little or no history of insecticide application.

During the past few years much has been written about the presence of various insecticide residues in our environment. It has been fairly well established that most components of the ecosphere contain detectable amounts of one or more of the organochlorine insecticides and the addition of these chemicals to the environment continues each year (Dustman and Stickel, 1969).

An increasing demand for soybean products has stimulated an expansion of acreages devoted to this legume in Alabama and other southeastern states. Various insect pests sometimes necessitate the use of chemical control agents for the successful production of a soybean crop. During the 1968 and 1969 growing seasons, such chemicals as DDT, toxaphene, carbaryl, methyl parathion, and parathion were applied to soybean fields in Alabama to control insect pests.

Many of these fields are located in areas supporting high populations of such game species as bobwhite quail, cottontail and swamp rabbits, and white-tailed deer. It was suspected that these animals, living in close association with these fields, would come in contact with some of the insecticides in their normal movements and feeding habits.

A study was initiated to determine the occurrence and magnitude of insecticide contamination in the above game species found in or adjacent to selected insecticide-treated soybean fields, since these species are important game animals and are eaten by many people who hunt them.

METHODS AND MATERIALS

Collection and Preparation of Samples. During the summers and autumns of 1968 and 1969, landowners in various Alabama counties were interviewed to determine if any insecticides had been applied to their soybean fields during the growing season. The kinds and amounts were determined as accurately as possible. Only those fields treated with one or more of the organochlorine insecticides and located in good game habitat were selected as study areas. The insecticide formulation most often used was a mixture of DDT, toxaphene, and methyl parathion (an organophosphate). Many fields had not been treated, according to the owners, and many others had been treated with carbaryl or methyl parathion, which present little or no persistent residue problem. Areas were also located that had little or no history of organochlorine insecticide treatment and these areas were designated as control areas.

Bobwhite quail from treated and control areas were hunted with dogs and collected by shooting with shotguns. Quail taken in this manner were kept whole and placed in ice-filled coolers for transporting to the Auburn campus.

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Table I. Organochlorine Insecticide Residues in Bobwhite Quail Collected from Treated Soybean Fields in Sumter and Bullock Counties, Alabama, During 1969

County	Quail number	Residue (ppm lipid basis) DDT	DDE	DDD	Total DDT	Toxaphene	Heptachlor epoxide
Sumter	1	1.80	5.40	*	7.20	T	*
	2	2.10	20.00	T	22.10±	10.30	*
	3	15.40	20.70	T	36.10±	88.90	*
	4	T[a]	4.40	*	4.40±	*	1.30
	5	5.80	12.80	T	18.60±	26.50	*
	6	*[b]	5.80	*	5.80	*	2.40
	7	2.10	12.10	*	14.20	17.40	*
	8	9.65	13.10	*	22.75	18.75	*
	9	*	4.70	*	4.70	*	*
	10	0.36	2.24	*	2.60	*	*
	11	T	9.20	*	9.20±	*	*
	12	T	5.65	*	5.65±	*	1.17
	13	*	2.16	*	2.16	*	*
	14	*	27.90	*	27.90	*	*
	15	*	18.50	*	18.50	*	*
	16	15.50	30.90	*	46.60	*	*
	17	16.00	23.90	5.70	45.60	*	*
Bullock	1	9.60	17.60	*	27.20	T	*
	2	0.76	1.31	T	2.07±	T	*
	3	*	18.50	*	18.50	*	*

[a] T = <0.01 ppm. [b] * = undetected at approximately 0.001 ppm level of sensitivity.

White-tailed deer and rabbits were shot at night with either a 0.243 caliber rifle or 12-gauge shotgun. Approximately 5 lb of edible meat was taken from each deer and placed in cardboard freezer boxes. Rabbits were kept whole. Samples of deer meat and the rabbits were transported in ice-filled coolers to freezers at the Auburn campus.

Laboratory preparation consisted of skinning, eviscerating, and removing head and feet from the quail. Meat was stripped from the carcasses and chopped to a fine consistency using a meat cleaver. Rabbits were skinned and pieces of edible meat were removed from the carcass and blended in a Waring blender. One-half pound subsamples of the meat taken from deer were also blended in a Waring blender. The finely chopped or blended samples were placed in $\frac{1}{2}$-pt ice cream cartons and stored in a freezer until analyzed.

Collections were made from Clarke, Escambia, Wilcox, Bullock, Elmore, and Sumter counties. Control animals were taken from areas in Bullock, Sumter, and Lee counties with little or no history of organochlorine insecticide treatment.

Extraction and Clean-Up Procedures. Extraction and clean-up procedures used in the study are described in Section 231 of the Pesticide Analytical Manual (1968). This is a general method for extraction and cleanup of fatty foods.

Considerations for analysis of toxaphene residues are described in Section 211.5D of the Pesticide Analytical Manual (1968).

Gas Chromatography. A Varian Aerograph Model 204 Gas Chromatograph equipped with a tritium electron capture detector was used for residue analysis. The gas chromatograph was connected to a Honeywell 15 recorder. Glass coil columns 5 ft in length × $\frac{1}{8}$ in o.d. packed with 5% DC 200 on Gas Chrom Q were employed in the analysis. All tests were completed using nitrogen as a carrier gas. Operating conditions were as follows: carrier flow, 40 ml/min; detector temperature, 185°; inlet temperature, 175°; column temperature, 165°; attenuation, 64×10^{-10}; and chart speed, 15 in. per hour. Injection of 4-μl quantities of extracts were made with a Hamilton 701 10-μl syringe. Operating at the above condition a minimum sensitivity level of 0.01 ppm (on lipid basis) was obtained for the insecticides of major interest.

Thin-Layer Chromatography. Thin-layer chromatography was used to verify the qualitative results in this study. The technique used was described in Chapter IV, Section 411, in the Pesticide Analytical Manual (1968).

RESULTS AND DISCUSSION

A total of 20 quail, 31 rabbits, and 22 deer was collected in or near selected insecticide-treated soybean fields in Alabama. Results of residue analyses are reported on a lipid basis, since Food and Drug Administration residue tolerance levels in fatty foods are based on the amount of residue in the fat. Twenty-one deer, 17 rabbits, and 17 quail were collected from the selected untreated areas. Insecticide residues detected in the 73 experimental and 55 control animals were DDT, DDE, DDD, toxaphene, dieldrin, and heptachlor epoxide. The only insecticide residues occurring with any degree of regularity were DDT and DDE. The results of the analysis for insecticide residues in bobwhite quail are presented in Table I. Bobwhite quail were found to contain considerably more DDT residue than either deer or rabbits (Tables II and III). Quail averaged 17.08± ppm (total DDT) as compared to 3.00± and 2.47± ppm for deer and rabbits, respectively. Computed averages exclude amounts detected in trace quantities.

Toxaphene was detected in 5 of the 20 quail from treated areas in amounts ranging from 10.30 to 88.90 ppm, with an average residue level of 32.24± ppm. The remaining 15 birds did not contain reportable amounts of toxaphene. Heptachlor epoxide was detected in 3 of the 20 samples in amounts of 1.11, 1.30, and 2.40 ppm. Toxaphene was detected in 2 of 31 test rabbits. One contained 1.20 ppm; the other contained 12.35 ppm. One rabbit contained a trace of heptachlor epoxide. Residues of toxaphene were detected in 3 of the 22 deer collected from treated soybean fields. They contained 1.70, 6.04, and 8.70 ppm, respectively. Heptachlor epoxide was not detected in any of the deer tested.

Table II. Organochlorine Residues Detected in White-Tailed Deer Collected from Treated Soybean Fields in Clarke, Escambia, Sumter, and Wilcox Counties, Alabama, During 1968–1969

County	Deer number	Residue (ppm lipid basis) DDT	DDE	DDD	Total DDT	Toxaphene
Sumter	1	*[b]	1.50	*	1.50	*
	2	T[a]	0.93	*	0.93±	*
	3	T	0.93	T	0.93±	*
	4	4.80	T	1.20	6.00±	*
	5	2.64	0.17	0.99	3.80	*
	6	2.29	T	1.25	3.54±	*
	7	3.55	0.96	0.99	5.50	*
	8	9.20	4.60	5.00	18.80	*
	9	0.89	1.44	0.83	3.16	*
	10	3.33	0.66	1.66	5.65	*
	11	1.56	0.94	*	2.50	*
	12	4.40	0.82	T	5.22±	*
	13	1.06	2.06	0.86	3.98	*
Escambia	1	*	*	*		
	2	0.60	T	*	0.60±	1.70
	3	*	*	*		*
	4	*	*	*		*
Wilcox	1	1.20	*	*	1.20	*
	2	*	*	*		6.04
	3	T	*	*	T	8.70
Clarke	1	1.60	*	0.41	2.01	*
	2	0.52	*	0.18	0.70	*

[a] T = <0.01 ppm. [b] * = undetected at approximately 0.001 ppm level of sensitivity.

Table III. Organochlorine Insecticide Residues Detected in Rabbits Collected from Treated Soybean Fields in Elmore, Sumter, Escambia, and Clarke Counties, Alabama, During 1968–1969

County	Rabbit number	Residue (ppm lipid basis) DDT	DDE	DDD	Total DDT	Toxaphene	Heptachlor epoxide
Elmore	Composite of 3	0.50	1.60	*	2.10	*	*
Sumter	Composite of 4	0.54	0.50	0.50	1.54	*	*
	5	*[b]	6.70	*	6.70	*	*
Escambia	Composite of 6	0.25	0.47	0.29	1.01	*	*
	7	1.91	1.46	1.27	4.64	*	*
	8	*	*	*		*	*
	9	*	1.35	*	1.35	12.35	*
	10	*	*	*		*	*
	11	0.50	0.50	*	1.00	*	*
	12	*	*	*		*	*
	13	*	0.33	*	0.33	*	*
	14	*	4.14	*	4.14	*	*
	15	0.50	1.66	0.66	2.82	*	*
	16	1.49	3.19	1.13	5.81	*	*
	17	*	*	*		*	*
	18	0.33	1.60	*	1.93	1.20	*
Clarke	1	3.20	1.70	0.60	5.50	*	T[a]
	2	0.88	4.60	1.40	6.88	*	T
	3	10.00	5.00	3.00	18.00	*	T
	4	1.04	0.94	0.32	2.30	*	T
	5	6.00	3.60	1.00	10.60	*	*

[a] T = <0.01 ppm. [b] * = undetected at 0.001 ppm level of sensitivity.

Analysis of all animals collected from areas of little or no history of insecticide treatment also showed DDT to be the only insecticide residue occurring regularly. The results are presented in Table IV. Again the bobwhite quail contained DDT residues generally higher than deer or rabbits. Toxaphene (1.26 ppm) was detected in a composite sample from three deer collected in Sumter County, Alabama, and trace amounts of dieldrin were detected in a composite sample of four rabbits collected in the Lee County, Alabama, area. These were the only two insecticide residues detected in the control animals other than DDT.

DISCUSSION

When reporting results such as those obtained in this study, one is challenged to explain the significance of insecticide residues in these game animals from the standpoint of their chronic effects upon the species and from a human health standpoint, since these animals are used for human consumption.

Accumulations of DDT in the food chains of certain species of birds, as well as accumulations within their bodies, have been reported to have serious effects upon the reproductive ability of these birds (Anderson and Hickey, 1969; Bitman, 1969; Hickey and Anderson, 1968; Porter and Wiemeyer, 1969). Most of the species in question were associated with aquatic food chains.

El Sayed *et al.* (1967) found DDT residues in the fat of certain bird species associated with Louisiana cotton fields. Total DDT residues ran as high as 170 ppm in a house sparrow (*Passer domesticus*).

Table IV. Organochlorine Insecticide Residues in Deer, Rabbits, and Quail Collected from Areas in Alabama During 1968–1969 with Little or No History of Insecticide Application

County	Species	Residue (ppm lipid basis) DDT	DDE	DDD	Total DDT	Toxaphene	Dieldrin
Sumter	Deer (composite of 3)	0.12	*[a]	*	0.12	*	*
	Deer (composite of 4)	0.43	0.12	0.25	0.80	*	*
	Deer (composite of 3)	0.65	0.61	*	1.26	1.27	*
	Deer (composite of 8)	T[b]	*	*	T	*	*
	Deer	*	*	*	*	*	*
Pike	Deer	*	*	*	*	*	*
	Deer	*	*	*	*	*	*
Sumter	Rabbit (composite of 6)	T	T	*	T	*	*
Lee	Rabbit (composite of 4)	*	T	*	T	*	*
Lee	Rabbit (composite of 3)	*	0.79	*	0.79	*	*
Lee	Rabbit (composite of 4)	*	*	T	T	*	T
Barbour	Quail	*	3.10	*	3.10	*	*
	Quail	*	1.90	*	1.90	*	*
	Quail	*	2.80	*	2.80	*	*
	Quail	*	0.71	*	0.71	*	*
	Quail	*	1.09	*	1.09	*	*
	Quail	*	2.40	*	2.40	*	*
	Quail	*	0.75	*	0.75	*	*
	Quail	*	0.60	*	0.60	*	*
	Quail	*	1.80	*	1.80	*	*
	Quail	*	1.20	*	1.70	*	*
	Quail	*	1.50	*	1.50	*	*
	Quail	*	0.55	*	0.55	*	*
	Quail	*	2.06	*	2.06	*	*
	Quail	*	1.60	*	1.60	*	*
	Quail	*	2.40	*	2.40	*	*
	Quail	*	1.30	*	1.30	*	*
	Quail	*	2.80	*	2.80	*	*

[a] * = undetected at 0.001 ppm level of sensitivity. [b] T = <0.01 ppm.

Seven bobwhite quail collected from selected environments in Louisiana contained residues of DDT and its metabolites ranging from 0.01 to 1.090 ppb (Epps *et al.*, 1967).

Residue levels in tissue samples that indicate either a hazardous level or a safe level have not been clearly defined in most species (Stickel, 1968). Azevedo *et al.* (1965) reported as much as 1280 ppm of DDT in the fat of pheasants that exhibited an 11% reduction in chick survival. Hunt and Keith (1963) reported DDT residues in the fat of pheasants ranging from 1542 to 3647 ppm in an area where reduced production was suspected.

While it is not advisable to compare different species in their response to insecticide residues, the average DDT contamination of bobwhite quail in this study is not as high as residues reported from other bird populations exhibiting lowered reproductive rates.

Published reports of organochlorine residues in mammalian species are not nearly as numerous as those concerning various bird species. However, mule deer (*Odocoileus hemionus*) collected near orchards in Colorado contained residues of DDT and its metabolites ranging from 0.4 to 2.8 ppm (Jewel, 1967). The renal fat of white-tailed deer collected from various South Dakota counties contained residues of DDT and metabolites averaging 0.2 ppm (Greenwood *et al.*, 1967). Following three successive years of application of 1 lb of DDT per acre to areas of Montana, Colorado, and New Mexico, mule deer were found to contain an average of 12 ppm (wet weight basis) of DDT and its metabolites in their fatty tissues (Pillmore and Finley, 1963).

Rabbits collected from seven localities in Louisiana contained residues of DDT and metabolites in amounts ranging from 0 to 0.220 ppm (Epps *et al.*, 1967). These residues were calculated using whole wet weights rather than fat alone.

Insecticide residues detected in the various species associated with treated soybean fields in Alabama are not appreciably different from residues detected in game species from other areas of the U. S.

The Food and Drug Administration has established insecticide residue tolerance levels for domestic meat and poultry. Smith and Mussehl (1964) and Mussehl and Finley (1967), in discussing the significance of DDT residues in game birds, refer to a statement from the FDA suggesting that insecticide-treated areas be closed to hunting until residues in game animals are not appreciably in excess of those tolerated in domestic meats.

Average amounts of DDT detected in the fat of deer and rabbits were well below the 7-ppm FDA tolerance level for DDT residues allowed in domestic meats. Residues of DDT detected in bobwhites associated with DDT-treated soybean fields were considerably higher than the residue level that would be tolerated by the FDA in commercial poultry.

No insecticide residue tolerance levels have been established for game animals. Consequently, within certain limits, hunting bans based on tolerances established for domestic meat and poultry do not seem realistic. It seems that meaningful tolerance limits should be established for game species that fully explain the difference between tolerance and safety levels. The game species tested in this study contain residues of chlorinated hydrocarbon insecticides in amounts much less than the investigators had suspected they might. There is presently no evidence to indicate that the levels of DDT detected in the animals analyzed in this study pose any hazard to people utilizing them as food.

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Fate of Zinc Phosphide and Phosphine in the Soil–Water Environment

H. Wayne Hilton* and William H. Robison

Zinc phosphide, mixed with three soils at five moisture levels, decomposed with the liberation of variable amounts of phosphine gas. Oxidation at the soil surface yielded zinc and phosphate ions as the ultimate products. In a closed system the soils slowly reabsorbed and oxidized the PH_3. Phosphine placed at high concentration in contact with the same soils oxidized slowly and incompletely to phosphate ion. The rate of zinc phosphide decomposition increased with increasing moisture; phosphine absorption decreased with added water. Soil types differed markedly in the ability to oxidize Zn_3P_2 or PH_3. The differences could not easily be related to known soil properties. Zinc phosphide did not decompose in water from streams, domestic source, or the ocean. Acids and bases hydrolyzed Zn_3P_2 to PH_3, but the reaction was not entirely pH dependent. A phosphate buffer of pH 7.00 extensively hydrolyzed Zn_3P_2 at room temperature.

Most of the zinc phosphide (Zn_3P_2) applied over sugarcane fields as a constituent of a rodenticide bait could be expected to reach the soil (Elmore and Roth, 1943; Hayne, 1951; Nass *et al.*, 1970). There appears to be no information on the fate of Zn_3P_2 in soil or in natural waters, nor on the fate of any phosphine gas (PH_3) liberated as a result of hydrolysis.

The gas chromatographic procedure for the measurement of PH_3 (Robison and Hilton, 1971) made it possible to determine PH_3 released from the incubation of Zn_3P_2 with soils and water, and to determine the fate of PH_3 placed in a closed system with the same substrates. Another study (Hilton and Mee, 1972) indicated that PH_3, as $^{32}PH_3$, reacted in part with strong acid, plant tissue, and soil to form nonvolatile water-soluble oxidation products. We designed the present experiments to measure the PH_3 resulting from Zn_3P_2 hydrolysis and the phosphate ion (as H_3PO_4) resulting from the oxidation of Zn_3P_2 or PH_3.

MATERIALS AND METHODS

Soils and Water Sources. Three oxide clay soils of volcanic origin represented 60 to 70% of the sugarcane topsoils in Hawaii: a Hydrol Humic Latosol (HHL) from the high rainfall climate on the island of Hawaii, pH 5.7, with a high organic matter content of 12 to 15%; a Humic Ferruginous Latosol (HFL) from a moderate rainfall windward Kauai location, pH 5.4, with an intermediate organic matter content of 5 to 8%; and a Low Humic Latosol (LHL) from a low rainfall area of Oahu and typical of soils on Maui and leeward Kauai, pH 6.9, with low organic matter content of 3 to 5%.

Water sources included the Wailua River (Kauai, pH 7.05), Grove Farm Co. irrigation water (Kauai, pH 7.20), Keapana Stream (Kauai, pH 7.65); Wailuku River (Hawaii, pH 7.52); Honolulu domestic water (Oahu, pH 7.90), laboratory-distilled water (pH 6.00), ocean water (Oahu, pH 8.20). Buffer solutions of pH 4.00 and 7.00 were also included.

Gas Chromatography. A Microtek MT 220 (Tracor Instruments, Austin, Texas), fitted with a flame photometric detector and a Melpar filter, isolated the 526-nm band specific for volatile phosphorus compounds. Phosphine gas generated from Zn_3P_2 and diluted to 1.00 ppm with N_2 served as a reference standard (Robison and Hilton, 1971; Berck *et al.*, 1970), with a sensitivity limit of 20 pg.

Operating conditions were: detector temperature, 150°, inlet temperature, 220°; column, 4 ft × 1/4 in. borosilicate glass packed with 5% QF-1 on Gas Chrom Q, 80 to 100 mesh, and heated isothermally at 48°; nitrogen carrier gas, 60 ml/min; detector gases, H_2 at 150 ml/min; air at 35 ml/min; and O_2 at 15 ml/min. Phosphine eluted 20 sec after injection; peak heights were linear with amounts of PH_3. There were no interferences and no detectable volatile compounds other than PH_3.

Zn_3P_2 Incubation with Soils. Zinc phosphide in amounts equivalent to 1000 ppm of P (4.17 mg of Zn_3P_2 per g of soil)

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