RESIDUE REVIEWS

VOLUME 59

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Residues of Pesticides and Other Contaminants in the Total Environment

Editor
FRANCIS A. GUNTHER

Assistant Editor
JANE DAVIES GUNTHER

Riverside, California

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本卷收入5篇论文：① 无控制植物生长的区域内，使用2、4、5T对鸟类安全性评价：认为在现在使用2、4、5T及其酯或盐类的浓度，不会对施药区内鸟类的卵、雏、成禽有急性或慢性中毒的影响。② 为适用气液色谱分析对除草剂残留物所作的化学性衍化处理：叙述了对易挥发、热稳定性差的除草剂，使用甲基化、卤代等方法，即可将残留物变成适于仪器分析的衍生物。③农药和其它有机污染的现场用萤光光谱法分析。④ DDT及其衍生物向大气中的移动：指出不同的加工方法可以影响DDT的挥发性，空气中的DDT主要来自喷洒过程并讨论了消失途径。DDT及衍生物在停用后很多年才能停止向大气中散布。⑤ DDT对高等动物繁殖的影响：分别叙述了DDT及其各种异构体与衍生物对哺乳动物、鸟类繁殖等方面的影响。

Foreword

Worldwide concern in scientific, industrial, and governmental communities over traces of toxic chemicals in foodstuffs and in both abiotic and biotic environments has justified the present triumvirate of specialized publications in this field: comprehensive reviews, rapidly published progress reports, and archival documentations. These three publications are integrated and scheduled to provide in international communication the coherency essential for nonduplicative and current progress in a field as dynamic and complex as environmental contamination and toxicology. Until now there has been no journal or other publication series reserved exclusively for the diversified literature on "toxic" chemicals in our foods, our feeds, our geographical surroundings, our domestic animals, our wildlife, and ourselves. Around the world immense efforts and many talents have been mobilized to technical and other evaluations of natures, locales, magnitudes, fates, and toxicology of the persisting residues of these chemicals loosed upon the world. Among the sequelae of this broad new emphasis has been an inescapable need for an articulated set of authoritative publications where one could expect to find the latest important world literature produced by this emerging area of science together with documentation of pertinent ancillary legislation.

The research director and the legislative or administrative advisor do not have the time even to scan the large number of technical publications that might contain articles important to current responsibility; these individuals need the background provided by detailed reviews plus an assured awareness of newly developing information, all with minimum time for literature searching. Similarly, the scientist assigned or attracted to a new problem has the requirements of gleaning all literature pertinent to his task, publishing quickly new developments or important new experimental details to inform others of findings that might alter their own efforts, and eventually publishing all his supporting data and conclusions for archival purposes.

The end result of this concern over these chores and responsibilities and with uniform, encompassing, and timely publication outlets in the field of environmental contamination and toxicology is the Springer-Verlag (Heidelberg and New York) triumvirate:

Residue Reviews (vol. 1 in 1962) for basically detailed review articles concerned with any aspects of residues of pesticides and other chemical contaminants in the total environment, including toxicological considerations and consequences.

Bulletin of Environmental Contamination and Toxicology (vol. 1 in 1966) for rapid publication of short reports of significant advances and discoveries in the fields of air, soil, water, and food contamination and pollution as well as methodology and other disciplines concerned with the introduction, presence, and effects of toxicants in the total environment.

Archives of Environmental Contamination and Toxicology (vol. 1 in 1973) for important complete articles emphasizing and describing original experimental or theoretical research work pertaining to the scientific aspects of chemical contaminants in the environment.

Manuscripts for *Residue Reviews* and the *Archives* are in identical formats and are subject to review, by workers in the field, for adequacy and value; manuscripts for the *Bulletin* are not reviewed and are published by photo-offset to provide the latest results without delay. The individual editors of these three publications comprise the Joint Coordinating Board of Editors with referral within the Board of manuscripts submitted to one publication but deemed by major emphasis or length more suitable for one of the others.

October 20, 1975 Coordinating Board of Editors

Preface

That residues of pesticide and other contaminants in the total environment are of concern to everyone everywhere is attested by the reception accorded previous volumes of "Residue Reviews" and by the gratifying enthusiasm, sincerity, and efforts shown by all the individuals from whom manuscripts have been solicited. Despite much propaganda to the contrary, there can never be any serious question that pest-control chemicals and food-additive chemicals are essential to adequate food production, manufacture, marketing, and storage, yet without continuing surveillance and intelligent control some of those that persist in our foodstuffs could at times conceivably endanger the public health. Ensuring safety-in-use of these many chemicals is a dynamic challenge, for established ones are continually being displaced by newly developed ones more acceptable to food technologists, pharmacologists, toxicologists, and changing pest-control requirements in progressive food-producing economies.

These matters are of genuine concern to increasing numbers of governmental agencies and legislative bodies around the world, for some of these chemicals have resulted in a few mishaps from improper use. Adequate safety-in-use evaluations of any of these chemicals persisting into our foodstuffs are not simple matters, and they incorporate the considered judgments of many individuals highly trained in a variety of complex biological, chemical, food technological, medical, pharmacological, and toxicological disciplines.

It is hoped that "Residue Reviews" will continue to serve as an integrating factor both in focusing attention upon those many residue matters requiring further attention and in collating for variously trained readers present knowledge in specific important areas of residue and related endeavors involved with other chemical contaminants in the total environment. The contents of this and previous volumes of "Residue Reviews" illustrate these objectives. Since manuscripts are published in the order in which they are received in final form, it may seem that some important aspects of residue analytical chemistry, biochemistry, human and animal medicine, legislation, pharmacology, physiology, regulation, and toxicology are being neglected; to the contrary, these apparent omissions are recognized, and some pertinent manuscripts are in preparation. However, the field is so large and the interests in it are so varied that the editors and the Advisory Board earnestly solicit suggestions of topics and authors to help make this international book-series even more useful and informative.

"Residue Reviews" attempts to provide concise, critical reviews of timely advances, philosophy, and significant areas of accomplished or needed endeavor in the total field of residues of these and other foreign chemicals in any segment of the environment. These reviews are either general or specific, but properly they may lie in the domains of analytical chemistry and its methodology, biochemistry, human and animal medicine, legislation, pharmacology, physiology, regulation, and toxicology; certain affairs in the realm of food technology concerned specifically with pesticide and other food-additive problems are also appropriate subject matter. The justification for the preparation of any review for this book-series is that it deals with some aspect of the many real problems arising from the presence of any "foreign" chemicals in our surroundings. Thus, manuscripts may encompass those matters, in any country, which are involved in allowing pesticide and other plant-protecting chemicals to be used safely in producing, storing, and shipping crops. Added plant or animal pest-control chemicals or their metabolites that may persist into meat and other edible animal products (milk and milk products, eggs, etc.) are also residues and are within this scope. The so-called food additives (substances deliberately added to foods for flavor, odor, appearance, etc., as well as those inadvertently added during manufacture, packaging, distribution, storage, etc.) are also considered suitable review material. In addition, contaminant chemicals added in any manner to air, water, soil or plant or animal life are within this purview and these objectives.

Manuscripts are normally contributed by invitation but suggested topics are welcome. Preliminary communication with the editors is necessary before volunteered reviews are submitted in manuscript form.

Department of Entomology F.A.G.
University of California J.D.G.
Riverside, California
October 20, 1975

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The evaluation of the safety of 2,4,5-T to birds in areas treated for vegetation control

By

EUGENE E. KENAGA*

Contents

I. Introduction

2,4,5-Trichlorophenoxyacetic acid (2,4,5-T) is marketed as a herbicide in the form of esters and salts. Some commonly used esters have been the isopropyl, butyl, isooctyl, butoxy ethanol (= butoxyethyl), and propyleneglycol butyl ether (PGBE) derivatives of 2,4,5-T. Some commonly used salts have been the dimethylamine, triethylamine, and the oleic-1,3-propylenediamine derivatives of 2,4,5-T.

2,4,5-T and derivatives are systemic herbicides applied for the control of woody and herbaceous weed plants. Dosages vary from 0.5 to 16 lb/A of 2,4,5-T in various habitats such as rights-of-way, range and pasture, rice, sugarcane, and turf (*Weed Science Society of America* 1974).

Data concerning the persistence of 2,4,5-T in grasslands in Texas was reviewed by BOVEY and BAUR (1972). They showed residues from 1 lb of 2,4,5-T/A treatments of 38 to 144 ppm of 2,4,5-T on grass immediately after application, which declined to less than 6 ppm within six weeks of treatment, at a steady rate of decrease. 2,4,5-T as the butoxyethyl derivative, when applied to grass by MORTON *et al.* (1967), was found to be present as a residue the first day in the forms of the ester and the acid,

* Dow Chemical U.S.A., Midland, Michigan 48640.

but not as the ester one week later, showing the ability of plants to readily deesterify this ester compound. Massive dosages (a total of 947 lb/acid equivalent) of 2,4,5-T as formulations (butyl esters mostly) applied over an eight-year period to soil were found to contain less than 0.1 ppm of 2,4,5-T one to two years after the last application (WOOLSON *et al.* 1972). This work indicates rapid degradation of 2,4,5-T in soil under the conditions of these tests. Microorganisms are believed to be the principal cause of decomposition of 2,4,5-T in soil (NORRIS *et al.* 1972).

LUTZ *et al.* (1973) showed very little downslope movement of 2,4,5-T at dosages up to 4 lb/A in a North Carolina watershed having an average slope of approximately 27%, indicating very little water transport. NORRIS and MOORE (1970) found that concentrations of 2,4,5-T did not exceed 1 ppm in streams adjacent to carefully controlled forest spray operations and that chronic entry of 2,4,5-T into streams for long periods of time after application did not occur.

KENAGA (1974) reviewed the stability of esters of 2,4,5-T in aquatic environments, finding them to be hydrolyzed within a matter of days in most kinds of water, except those which are highly acidic. Highly acid natural waters are not common. The degradation of 2,4,5-T itself in water is hastened by sunlight. Fish also were found to rapidly hydrolyze esters of chlorophenoxy alkanoic acids.

The above references, by no means complete, indicate 2,4,5-T and esters may be degraded by physical, chemical, and biological means. Microorganisms, plants, and animals appear to degrade 2,4,5-T and its derivatives, sometimes very rapidly. Residue and toxicological background information is necessary for safety evaluations of compounds in various segments of the environment. Birds live on or visit nearly all segments of the environment.

This review summarizes the toxicological effects of 2,4,5-T and derivatives to birds and the residues caused by application of 2,4,5-T under use conditions in order to match them up for the purposes of safety evaluation.

II. Toxicity and residues

The toxicity of 2,4,5-T and derivatives to birds has been studied by means of acute oral dosages, repeated daily oral dosages, dietary feeding to chick and adult birds, and external applications to eggs. The dietary feeding and egg application tests simulate most closely the most practical ways by which birds may be exposed to 2,4,5-T applications. Laboratory tests allow more precise answers to definitive questions than field tests, but field tests also take in parameters of weather, habits and habitats of the bird, and other factors not taken into account by laboratory tests; thus, both types of tests are desirable. The most informative tests are those in which residues of the pesticide or representative bird food are compared with lethal dietary toxicity concentrations.

a) Acute oral toxicity tests (LD_{50} determinations)

TUCKER and HUDSON (1970) administered single oral dosages to mallard (*Anas platyrhynchus*) drakes at concentrations of up to 2,000 mg/kg of 2,4,5-T (acid) with no mortality 17 to 19 days after treatments. Symptoms of intoxication following treatment appeared in 20 min and disappeared in one day. No gross pathological changes attributable to 2,4,5-T were observed in tissues of sacrificed survivors, nor was there any weight loss at the end of the 17 to 19 day observation period.

ROWE and HYMAS (1954) determined the acute oral LD_{50}'s of 2,4,5-T, and several derivatives in formulations using the chicken (*Gallus domesticus*). LD_{50} values for the formulations varied from 2,000 to 4,000 mg/kg while 2,4,5-T (acid) was 300 mg/kg.

WHITEHEAD and PETTIGREW (1972) reported the mortality from single oral dosages of 250, 400, 600, and 900 mg of 2,4,5-T/kg to four-week-old chicks of a broiler strain to be 0, 0, 33, and 40%, respectively. All of these high dosages caused the birds to stop eating and drinking for one to two days after treatment and to lose up to 10% of their body weight for three to four days after which normal weight growth rates resumed.

The LD_{50} values (see Table I) indicate that 2,4,5-T and the formulations tested are moderate to low in acute oral toxicity to birds.

Table I. *Acute oral toxicity of 2,4,5-T and formulations to birds.*

Species of bird	Form of 2,4,5-T	LD_{50} (mg/kg)	Reference
Mallard (adult)	Acid	> 2,000	TUCKER & HUDSON (1970)
Chicken (4-week-old broiler strain)	Acid	> 900	WHITEHEAD & PETTIGREW (1972)
Chicken (New Hampshire Red, male and female chicks)	Acid	310	ROWE & HYMAS (1954)
	BRUSH KILLER T[b]	4,000	ROWE & HYMAS (1954)
	BRUSH KILLER 50–50[a]	2,000	ROWE & HYMAS (1954)
	ESTERON® BRUSH KILLER (new)[c]	2,000	ROWE & HYMAS (1954)

[a] 27.2% butyl esters of 2,4-D and 26.5% butyl esters of 2,4,5-T.
[b] 52.2% butyl esters of 2,4,5-T.
[c] 34.8% mono-, di-, and tripropyleneglycol butyl ether esters of 2,4-D and 33.0% mono-, di-, and tripropyleneglycol butyl ether esters of 2,4,5-T.

b) Repeated daily oral toxicity tests

PALMER and RADELEFF (1969) and PALMER (1972) used a pipette to apply daily oral doses for ten days with various formulations and derivatives of 2,4,5-T to chickens. Dosages which caused approximately 50% mortality and no lethal effects and those causing no weight loss are shown in Table II. These doses do not necessarily simulate equivalent doses

Table II. *Mortality of weight loss of chickens fed various derivatives of 2,4,5-T with one dose/day for ten days.*

Salt or ester derivative of 2,4,5-T (formulation)	Dosage (mg/kg[a])/day for ten days			Reference
	LD_{50}	No mortality	No wt loss[b]	
Triethylamine salt (VEON® 245)	> 250–< 375	25	50	PALMER (1972)
2-Ethylhexyl ester (BRUSH RHAP®)	> 500	250	50	PALMER (1972)
Propyleneglycol butyl ether esters (ESTERON® 245 O.S.)	> 500	250	100	PALMER & RADELEFF (1973)

[a] Based on a dietary intake of 10% of body wt/day, the ppm equivalent would be 10× that of the mg/kg (*i.e.*, 250 mg/kg/day = 2,500 ppm in the diet).
[b] Compared to control.

consumed in the normal daily diet of the animals; however, all the dosages quoted represent higher dietary intake than would normally be encountered by birds on areas treated with 2,4,5-T.

c) Dietary feeding tests

1. Chicken tests.—The toxicity of 2,4,5-T, butoxyethyl ester (also known as butoxyethanol ester), to chickens was studied by WHITEHEAD and PETTIGREW (1972). Dietary concentrations of up to 100 ppm of 2,4,5-T acid equivalent (a.e.) were consumed over a three-week period with no mortality, no adverse effects on weight gain, growth rate, and no adverse findings from necropsy. At 5,000 and 7,500 ppm reduced feeding and 90 and 100% mortality occurred, respectively (see Table III). Chicks could tolerate up to 5,000 ppm for one week and resumed normal growth when returned to uncontaminated food. The birds were able to discriminate between treated and untreated food, rejecting the former when given a choice. The calcium and magnesium levels in the plasma of two-week-old chicks fed 5,000 ppm of 2,4,5-T for one week were not affected.

BJÖRKLUND and ERNE (1971) studied the effects of 2,4,5-T (in the form of the triethanolamine salt) on five-day-old chicks at the rate of 1,000 ppm acid equivalent in their drinking water for up to 201 days;

Table III. *Toxicity of 2,4,5-T to birds in dietary feeding studies.*

Form of 2,4,5-T[a]	Bird species	No. of days fed	ppm[a] fed	Effect	Reference
Acid	Bobwhite	5	2,776	LC_{50}	Fink (1974 b)
Acid	Mallard	5	4,640	10% mortality	Fink (1974 a)
Butoxyethanol ester	Japanese quail	5	> 5,000	LC_{50}	Heath *et al.* (1972)
	Ring-necked pheasant	5	3,950	LC_{50}	
	Mallard	5	5,000	10% mortality	
Acid	Chicken	7	2,000	0% mortality	Kenaga (1973)
n-Butyl ester	Chicken	7	500	0% mortality	
Isoctyl ester	Chicken	7	500	0% mortality	
Propyleneglycol butyl ether ester	Chicken	7	500	0% mortality	
Butoxyethyl ester	Chicken	14	7,500 (a.e.)	100% mortality	Whitehead & Pettigrew (1972)
	Chicken	21	5,000 (a.e.)	90% mortality	
	Chicken	21	2,000, 1,000 (a.e.)	0% mortality, reduced feeding and growth	
	Chicken	21	100, 10 (a.e.)	0% mortality	
Unnamed ester	Chicken	56	510 (a.e.)	0% mortality	Andersson *et al.* (1962)
Triethanolamine salt	Chicken	201	1,000(a.e.)[b]	100% mortality[c]	Björklund & Erne (1971)
		33	1,000(a.e.)[b]	27% mortality[c]	

[a] Active ingredient (a.i.) except where noted as acid equivalent (a.e.) in diet.
[b] Concentration in drinking water.
[c] See Table V for other effects.

chickens apparently consume 25 to 35% of their weight in water/day during the first few weeks of their lives and level off at about 20% before seven weeks of age (MERCK 1967). At these usage rates, 1,000 ppm of 2,4,5-T in the birds' dietary water is equivalent to an intake of 250 to 350 mg/kg/day initially and about 200 mg/kg/day over the latter part of the test. A concentration of 1,000 ppm of 2,4,5-T (a.e.) in the drinking water produced 27% mortality in chickens exposed from days 5 to 33, which establishes that the dosages were in the lethal range (see Table III). 2,4,5-T in the lethal range caused kidney enlargement and lesions in chickens during most of the test period apparently due to hypertrophy of the proximal convoluted tubular epithelium, but the kidneys began to return to the same size as the control after 201 days of exposure. Ultrastructural kidney changes induced by 2,4,5-T appeared to be reversible. The livers were smaller than average throughout the early exposure, but returned to larger than control size after 201 days' exposure (see Table IV). This may be an effect similar to the enhanced elimination of 2,4-D noted by BJÖRKLUND and ERNE (1966) in latter stages of the tests.

Table IV. *Effects of 2,4-D and 2,4,5-T on body, kidney and liver weights and kidney-liver weight ratios of chickens* (BJÖRKLUND and ERNE 1971).

Treatment	Exposure (days)	Average			
		Body wt (g)	Organ wt		Kidney-liver ratio
			Kidney (g)	Liver (g)	
2,4,5-T 1,000 ppm[a] in water from 5th day of life	14–18	57.5	3.00	2.61	1.15
	28	128	5.70	4.89	1.17
	104	965	23.2	30.45	0.81
	141–143	1,423	18.6	26.63	0.70
	201	1,767	15.4	41.90	0.37
Control	14–16	150	1.68	4.63	0.37
	28	347	3.90	9.85	0.41
	104	1,609	12.0	25.1	0.48
	141–143	1,843	10.4	34.5	0.30
	201	2,277	11.2	31.8	0.36

[a] 1,000 ppm of 2,4,5-T, acid equivalent, in the form of triethanolamine salt.

Residues of 2,4,5-T in various tissues of chickens drinking 1,000 ppm (a.e.) in their water for 104 days are shown in Table V. Highest residues occurred in the stomach and intestine (18 to 85 ppm). Small residues of 0.7 to 2.1 ppm were found in egg yolk. Residues in lung, kidney, and liver tissues were similar (3 to 6 ppm).

Dietary tests with 2,4,5-T and three ester derivatives, using White Leghorn chicks infested with coccidiosis, showed no mortality in seven days from the highest dosages tested (2,000 ppm of 2,4,5-T, and 500 ppm

Table V. *Residues of 2,4,5-T in various tissues of chickens fed 1,000 ppm[a] in their drinking water for 104 days* (BJÖRKLUND and EANE).

Tissues tested	2,4,5-T (ppm[b])
Liver	2.8
Kidney	5.9
Lung	6.1
Stomach	85.0
Small intestine	18.0
Egg yolk	0.7–2.1

[a] 1,000 ppm of 2,4,5-T, acid equivalent, in the form of the triethanolamine salt.
[b] Fresh weight.

each of the *n*-butyl, isooctyl, and propyleneglycol butyl ester (PGBE) esters of 2,4,5-T (KENAGA 1973) (see Table III).

2. Wild game bird tests.—FINK (1974 a) fed mallard ducklings concentrations of 215, 464, 1,000, 2,150, and 4,640 ppm of 2,4,5-T for five days in their total diet and held them three additional days on untreated food before making mortality counts. No mortality occurred in the treatments or controls except for 10% mortality at the 4,640 ppm treatment level. The LC_{50} value was thus $>$ 4,640 ppm. A reduction in feed consumption was noted for birds given the 2,150 and 4,640 ppm dietary treatments. No other symptoms of toxicity or behavioral abnormalities were noted at the dosage levels tested. The technical sample of 2,4,5-T used in these studies was found to contain 0.06 ppm of 2,3,7,8-tetrachlorodibenzo-*p*-dioxin (TCDD), an impurity which is present in current commercial 2,4,5-T products at $<$ 0.1 ppm.

FINK (1974 b) fed bobwhite (*Colinus virginianus*) chicks concentrations of 215, 464, 1,000, 2,150, and 4,640 ppm of 2,4,5-T for five days in their diets and held them three additional days on untreated food before making mortality counts. No effects were seen on birds consuming the 215, 464, and 1,000 ppm levels. At 2,150 ppm 20% mortality and some reduction in food consumption occurred. At 4,640 ppm 90% mortality and marked effects on body weight and food consumption were noted. The calculated LC_{50} was 2,776 ppm (95% confidence limits 2,084 to 3,698 ppm). Symptoms of toxicity preceding death were depression, wing droop, and loss of righting reflex. The technical sample of 2,4,5-T used for these tests was the same as in the mallard studies by FINK (1974 a) (see Table III).

HEATH *et al.* (1972) studied the toxicity of the butoxy-ethanol ester of 2,4,5-T to the young of the Japanese quail (*Coturnis coturnix japonica*), ring-necked pheasant, (*Phasianus colchicus*), and mallard. The LC_{50}s in

five-day dietary feeding studies eight days after the test started were all greater than 3,000 ppm (see Table III).

FINK (1974 c) conducted a "one-generation reproduction study" on bobwhite using technical 2,4,5-T containing 0.06 ppm of TCDD. Adult birds in 12 pens/treatment, each pen containing a male and two females, received 2,4,5-T *ad libitum* in their "game bird breeder" diet at treatment levels of 0, 5, and 50 ppm over an 18-week period. During the last eight weeks of the treatment eggs were collected daily and set for incubation weekly. The eggs took 21 days to hatch and the hatched chicks were observed for an additional 14 days while being maintained on untreated feed.

Data on the following parameters are tabulated as follows: body weight and food consumption (Table VI); eggs laid, eggs cracked, eggs

Table VI. *Body weight and food consumption data in bobwhite reproduction tests* (FINK 1974 c).

	2,4,5-T conc in diet (ppm)					
	Controls[a]		5		50	
Week	B.W. (g)[b]	F.C. (g)[c]	B.W. (g)[b]	F.C. (g)[c]	B.W. (g)[b]	F.C. (g)[c]
0	187	—	197	—	209	—
2	—	15	—	14	—	14
4	—	14	—	14	—	14
6	—	16	—	17	—	17
8	183	18	201	20	205	19
10	—	16	—	18	—	18
12	—	23	—	24	—	21
14	—	20	—	25	—	23
16	—	21	—	26	—	23
18	174	22	190	27	201	25

[a] The differences between controls and treatments were not statistically significant ($p < 0.05$).

[b] The body wt (B.W.) data are presented as a group mean.

[c] The food consumption (F.C.) data are presented as the group mean feed consumed/bird/day.

set, viable embryos, live three-week-old embryos, normal hatchlings, and 14-day-old survivors (Table VII); eggs laid/hen in eight weeks, % eggs cracked of eggs laid, % viable embryos of eggs set, % live three-week embryos of embryonated eggs, % normal hatchlings of live three-week embryos, % 14-day-old survivors of normal hatchlings, and 14-day-old survivors/hen (Table VIII); and eggshell thickness (Table IX).

Evaluation of the reproductive data demonstrates that no reproductive impairment resulted from consumption of 5 ppm or 50 ppm of 2,4,5-T in the food of the adult bobwhite over an 18-week feeding period.