Table III. Persistence of Parathion in Autoclaved and Nonautoclaved Acid Sulfate Soil under Flooded Conditions

	Parathion recovered, μg/20 g of soil	
Incubation, days	Autoclaved	Nonautoclaved
0	251	251
14	163	80
28	155	24

Table IV. Degradation of *p*-Nitrophenol by a *Bacillus* sp. Isolated from Parathion-Amended Alluvial Soil

	p-Nitrophenol recovered, ppm	
Incubation, hr	Inoculated	Uninoculated
0	12.3	14.3
24	3.0	13.0
48	0	13.5

centration in the soil decreased after 24 hr. In soils inoculated with autoclaved enriched culture, no appreciable degradation of parathion occurred during the 120-hr incubation period; also *p*-nitrophenol was not detected. Lichtenstein and Schulz (1964) reported that degradation of parathion in nonflooded soils proceeded either by hydrolysis or by reduction of the nitro group, depending on the microbial population. Hydrolysis of parathion by resting cells and cell-free extracts of a *Flavobacterium* sp., isolated from diazinon-amended rice fields, has been demonstrated recently (Sethunathan and Yoshida, 1972).

Bacterial Degradation of *p*-Nitrophenol. *p*-Nitrophenol formed by hydrolysis from parathion is toxic and its accumulation in soils might cause a pollution hazard. The fall in its concentration in the soil (Figure 1) indicated that it is rapidly broken down in flooded soils. A bacterium was isolated from flooded alluvial soil enriched by repeated additions of parathion. The bacterium identified as *Bacillus* sp. was tested for its ability to decompose *p*-nitrophenol. The bacterium readily decomposed *p*-nitrophenol (Table IV), but no other metabolite could be detected in the thin-layer chromatogram.

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Total Toxic Aldicarb Residues in Weeds, Grasses, and Wildlife from the Texas High Plains Following a Soil Treatment with the Insecticide

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Aldicarb residues in weeds, grasses, and wildlife following a soil application of the granular insecticide in dryland and irrigated fields were investigated by a gas chromatographic-flame photometric detector (gc-fpd) analysis. Residues of aldicarb and/or its metabolites (as the sulfone) were detected in 80% of the grass and weeds collected from treated areas and in 83% of samples from untreated sections of these fields. No detectable residues were found in samples from adjacent untreated, noncultivated areas outside the treated fields. In irrigated fields residues were detected in approximately 73% of samples from treated areas, 38% of samples from untreated sections of treated fields, and 31% of samples from adjacent untreated, noncultivated areas outside the treated fields. Residues in grasses and weeds gradually decreased with time and with plant growth, which created a dilution factor. Residues were detected in only one of the wildlife samples collected in and around the treated fields. This was an oriole showing 0.07 ppm of aldicarb and/or metabolites.

During 1971 a 10% granular formulation of aldicarb, 2-(methylthio)propionaldehyde *O*-(methylcarbamoyl)oxime, also known as UC-21149 and Temik, was tested as a broad spectrum insecticide for the control of a variety of cotton insects. In previous investigations by Andrawes *et al.* (1971), ^{14}C-labeled aldicarb residues remaining in the soil from an earlier experiment were found to translocate into crabgrass, tomato, and potato plants. This work was not pursued, however, and additional data on persistence and translocation were necessary. Further information was also needed to determine if residues appeared in birds and mammals which may feed on seeds and plants in the treated areas.

This report deals with aldicarb residues found in weeds, grasses, and wildlife collected in and adjacent to areas receiving a soil application of the 10% granular insecticide (aldicarb 10G) at a rate of 15 lb/acre (1.5 lb actual) in dryland and irrigated fields.

EXPERIMENTAL SECTION

Type of soil, application rate, and method of application were described in a previous paper (Woodham *et al.*,

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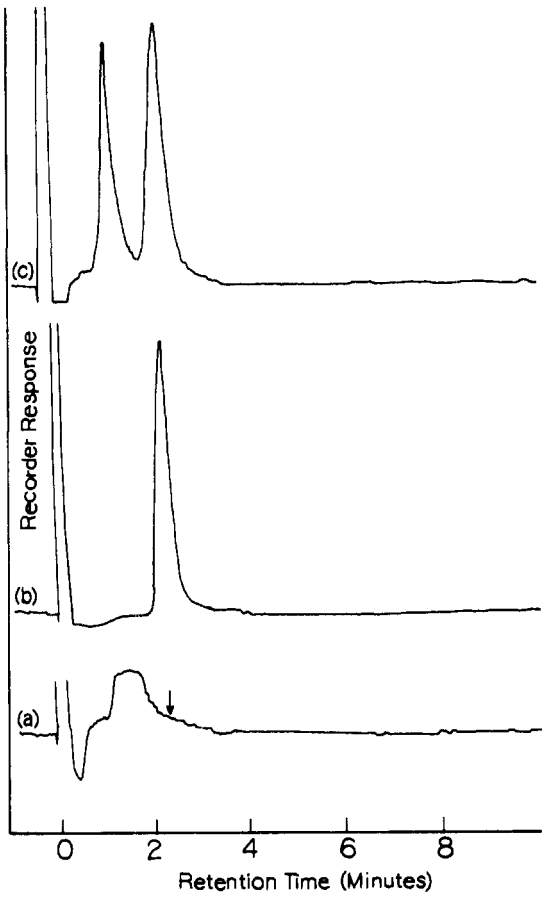

Figure 1. Gas chromatographic spectra of: (a) untreated sample of weeds after oxidization; (b) standard of 10.46 ng of aldicarb after oxidization; and (c) blank weed sample fortified with 0.11 ppm of aldicarb on a Carbowax column after oxidization. See gas chromatographic analysis section for complete operating parameters.

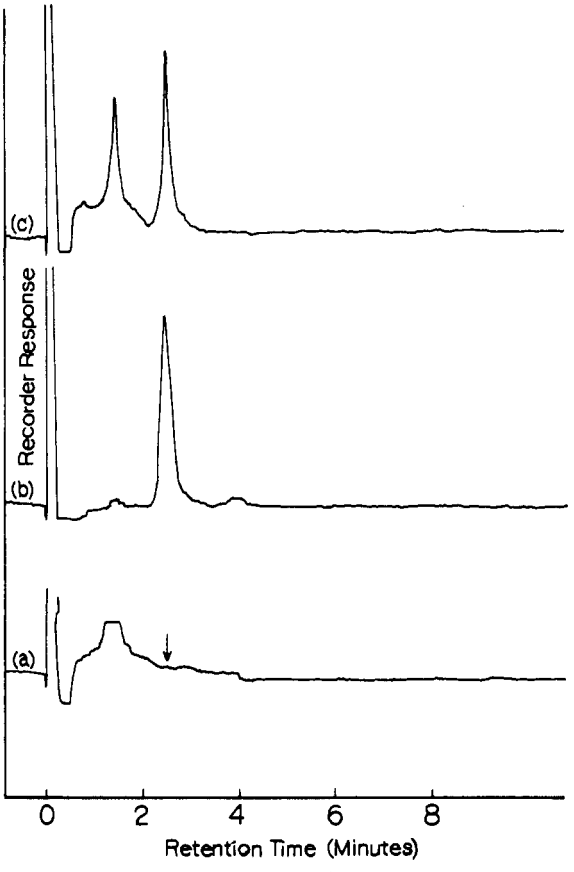

Figure 2. Gas chromatographic spectra of: (a) untreated biological sample after oxidization; (b) standard of 10.42 ng of aldicarb after oxidization; and (c) blank biological sample fortified with 0.12 ppm of aldicarb after oxidization, all on a Carbowax column. See gas chromatographic analysis section for complete operating parameters.

1973) reporting aldicarb residues in soil, cottonseed, and cotton lint.

Sampling Procedures. In most cases, samples of animals and birds were collected where found, usually within one-half mile of a treated field. Birds were killed with a shotgun as near a treated field as possible. Animal carcasses were placed in traps around watermelon fields for capturing live coyotes. No evidence of mortality in the animal or bird population was observed in the treated or adjacent areas.

Grass and weed samples were collected from treated fields according to the following plan. An imaginary line was drawn at two locations in each field. The line extended across the treated rows into the untreated portion of the field and outside the field into adjacent noncultivated, untreated areas. The imaginary line extended an equal distance into the untreated portion and outside the field. The average treated band width was 16 rows. Weeds were clipped, with aerial portions and roots being packaged separately for some samples; the entire plant was collected for the remaining samples. Samples were also collected from the two check fields at the same time. Similar type weeds and grass were sampled from the treated, untreated, and check plots. All samples were frozen as soon as possible and then shipped to the laboratory.

Analytical Procedures. The analytical methods of Woodham and Reeves were utilized with certain modifications necessary due to the type of sample materials involved. Confirmation of larger peaks in grasses and weeds and the one bird sample was confirmed by tlc.

Extraction. Grass and weed samples were composited according to sampling data and location, chopped thoroughly in a Hobart food chopper, and extracted according to the previously reported procedure for extraction of cottonseed samples with the following changes. Two-hundred milliliters of the acetone–water mixture was used to macerate the sample for 2 min in the blender. The blender jars were rinsed with 300 ml of the solvent mixture into half-gallon Mason jars for extraction. Aliquots of 350 ml were collected after filtration.

Birds and Mammals. Small mammals were processed whole; coyotes were dissected and certain tissues (stomach and liver) were selected for analysis; feathers were removed from birds and processed whole. Representative samples were extracted as described for the grass and weed samples, except that the samples were macerated with 150 ml of ACS grade acetone and transferred to the jars with two 75-ml portions of acetone, 100 g of anhydrous sodium sulfate was added, and 250-ml aliquots were collected.

Oxidization. *Grass and Weeds.* The same oxidization procedure as described previously by Woodham and Reeves was utilized for these samples, except that 5 ml of the peracetic acid was used. Fifty milliliters of the aqueous sodium bicarbonate solution was used to neutralize the acid. Residues in the flasks (following the evaporation step) were redissolved in 50 ml of chloroform.

Birds and Mammals. Wildlife samples were oxidized in essentially the same manner with the following changes. The samples were oxidized with 10 ml of the peracetic acid. One-hundred-and-fifty milliliters of the aqueous sodium bicarbonate solution was used to neutralize the excess acid. The oxidized extracts were transferred into 1000-ml separatory funnels, the flasks were rinsed with two 250-ml portions of chloroform through the filters into

Table I. Total Toxic Aldicarb Residues in Weeds and Grasses from Dryland Areas of the Texas High Plains (1971)

Field no.	Sampling interval, days	Sampling location	Residue, ppm,[a,b] as aldicarb sulfone
1	7	Untreated[c]	0.02
1	7	Outside[d]	<0.01
1	7	Treated[e]	42.80
1	29	Untreated	2.37
1	29	Outside	<0.01
1	29	Treated	0.96
1	29	Outside	<0.01
1	29	Untreated	2.85
4	43	Treated	10.40
4	43	Outside	<0.01
4	43	Treated	<0.01
4	43	Untreated	0.27
4	43	Outside	<0.01
4	43	Treated	2.75
4	42	Outside	<0.01
4	43	Untreated	19.64
7	Check		<0.01
7	Check		0.01
7	Check		<0.01
7	Check		<0.01
7	Check		<0.01
7	Check		<0.01

[a] Corrected for recovery from fortified samples. [b] Lower limits of detection = 0.01 ppm. [c] "Untreated" samples were collected from cultivated areas receiving no insecticide treatment, but within the treated field. [d] "Outside" samples were collected from noncultivated, untreated fields adjacent to fields receiving the insecticide treatment. [e] "Treated" samples were collected from the rows receiving Aldicarb treatment.

the funnels, and the filters were finally rinsed with 100 ml of fresh chloroform. The aqueous extracts were extracted with two additional 100-ml portions of fresh chloroform. After evaporating the chloroform, the residues were redissolved in 100 ml of a 1:1 (v/v) mixture of benzene–diethyl ether.

Florisil Cleanup. *Weeds and Grass.* Florisil cleanup of the extracts was performed as described in the previous method by Woodham and Reeves for the cleanup of cottonseed extracts. The elution sequence was identical except that 50 ml of 2% acetone–98% diethyl ether mixture was used for the second elution.

Biological Tissue. The chromatographic cleanup step for birds and mammals was identical to the procedure for weeds and grass with the following exceptions. The Florisil was prewet with 15 ml of a 1:1 (v/v) benzene–diethyl ether solvent mixture. The columns were eluted the second time with 100 ml of a 4% acetone–96% diethyl ether mixture. The third elution, containing the aldicarb sulfone, was made with the 1:1 benzene–diethyl ether solution. This eluate was evaporated to dryness as described previously and transferred to centrifuge tubes with benzene and stored for subsequent gc–fpd analysis. Gas–liquid chromatographic analyses were performed as described in the previously reported method by Woodham and Reeves for cottonseed and lint.

A series of control samples consisting of (1) solvent check, (2) untreated sample material and (3) aldicarb and aldicarb sulfoxide fortified sample materials were carried through the procedure with the unknown samples. Average recovery values for the fortified weeds and grass were 91.2% for the aldicarb and 88.3% for the sulfoxide; for birds and mammals, average values of 94.5% for the aldicarb and sulfoxide were obtained. All residues were corrected for recovery of aldicarb. No interfering peaks were detected in the solvents, reagents, or blank sample material. Lower limits of sensitivity were determined to be 0.01 ppm.

Table II. Total Toxic Aldicarb Residues in Weeds and Grasses from Irrigated Fields in the Texas High Plains (1971)

Field no.	Sampling interval, days	Sampling location[a]	Residue, ppm,[b,c] as aldicarb sulfone
2	23	Outside	<0.01
2	23	Untreated	<0.01
2	54	Treated	2.09
2	54	Outside	<0.01
2	54	Untreated	<0.01
2	54	Untreated	<0.01
2	54	Untreated	0.12
2	54	Outside	<0.01
2	54	Treated	0.03
2	54	Outside	<0.01
3	54	Outside	0.01
3	54	Untreated	23.59
3	74	Treated	<0.01
3	74	Outside	<0.01
3	74	Untreated	<0.01
3	74	Outside	<0.01
3	74	Outside	<0.01
3	74	Untreated	<0.01
3	74	Treated	2.94
5	51	Untreated	0.01
5	51	Untreated	<0.01
5	51	Treated	1.85
5	51	Untreated	0.55
5	51	Outside	0.03
5	51	Outside	<0.01
5	51	Treated	0.31
6	51	Untreated	0.02
6	51	Treated	0.43
6	51	Treated	<0.01
6	51	Treated	0.16
6	51	Untreated	<0.01
6	51	Outside	<0.01
6	51	Untreated	<0.01
6	51	Outside	0.02
6	51	Treated	<0.01
6	51	Treated	3.37
6	51	Outside	0.07
8	Check		<0.01
8	Check		<0.01
8	Check		0.01
8	Check		<0.01
8	Check		<0.01

[a] Refer to Table I for definitions of "treated," "untreated," and "outside" areas. [b] Corrected for recovery from fortified samples. [c] Lower limits of detection = 0.01 ppm.

RESULTS

Figure 1 depicts chromatographic tracings obtained from: (a) untreated weed and grass sample; (b) aldicarb sulfone standard (10.46 ng); and (c) untreated weeds and grass sample fortified with 0.11 ppm of aldicarb and carried through the complete procedure.

Figure 2 shows chromatographic tracings from: (a) untreated biological tissue; (b) aldicarb sulfone standard (10.42 ng); and (c) untreated biological tissue fortified with 0.12 ppm of aldicarb and subjected to the extraction, cleanup, oxidization, and glc analytical procedures described previously.

Table I presents residue data on the accumulation and disappearance of aldicarb and metabolites for weeds collected from treated dryland soil at various intervals after soil application of this pesticide.

Residues as high as 42.80 ppm were detected in a composite sample of nightshade, ironweed, and careless weed collected from a treated area of a dryland field 7 days following treatment, decreasing to 0.96 ppm 29 days after treatment.

Residues were also detected in untreated sections of dryland fields approximately 12–13 ft from the treated rows. Seven days following treatment, residues of 0.02 ppm were detected in a sample collected approximately 13 ft from treated rows. One sample of careless weed collected *ca.* 3 ft from treated rows showed a residue of 2.37 ppm 29 days after treatment; another sample of careless weed collected 13 ft from treated rows showed 2.85 ppm of aldicarb and/or metabolites. One thistle sample collected approximately 13 ft from the treated area showed a residue of 19.64 ppm. No detectable residues were found in untreated, noncultivated areas adjacent to the treated fields.

Table II presents residue data for samples of weeds and grass collected from treated fields receiving irrigation. Highest residues detected from treated areas were 2.09 ppm in a sample of careless weed 54 days after treatment, 2.94 ppm in a composite sample of Johnsongrass and ironweed 74 days following treatment, 1.85 ppm in a composite sample of careless weed, Johnsongrass, and Colorado grass, and 3.37 ppm in a composite sample of Johnsongrass and ironweed. Samples collected from untreated areas of the fields showed significant residues in only three samples: 0.12 ppm in a sample of nightshade collected 62 days after application; 23.59 ppm in a composite sample of careless weed and Johnsongrass collected 54 days following treatment; and 0.55 ppm in a sample of careless weed collected 51 days after treatment. All were collected approximately 13 ft from treated areas. Detectable residues were found in only three samples from adjacent untreated, noncultivated areas (0.03, 0.02, and 0.01 ppm) in material collected 51, 51, and 54 days, respectively, after treatment.

Residues were detected in only one wildlife sample, an oriole bird with 0.07 ppm of aldicarb and/or metabolites.

DISCUSSION

Aldicarb was found to be a pesticide which translocated into grasses and weeds in treated and untreated dryland and irrigated fields within a matter of weeks following treatment. No significant movement of the pesticide into uncultivated areas adjacent to either dryland or irrigated fields was noted. No indication of significant introduction into the biological food chain was noted since only one bird of the 14 sampled showed detectable residues; no detectable residues were found in any of the eight animals sampled.

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Mercury and Methylmercury Content of Agricultural Crops Grown on Soils Treated with Various Mercury Compounds

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Beans, cabbage, carrots, millet, onions, potatoes, and tomatoes were grown on silt loam, gravelly loam, and muck soils treated with 1 and 10 ppm of mercuric chloride, methylmercury dicyandiamide (PAN), or phenylmercuric acetate. Appreciable concentrations of methylmercury were present only in PAN-treated soils and in beans, millet, and tomatoes grown on those soils. Total mercury was usually less than 0.1 ppm in the edible plant portions, with the highest concentrations occurring most generally when growth occurred on the gravelly loam treated with PAN. Onion bulbs absorbed up to 1.1 ppm of total mercury. The highest concentrations of total mercury in plant stems and leaves were attained in potatoes and tomatoes.

The recently discovered ubiquitous presence and consequences of mercury in the environment have been amply reviewed (Goldwater, 1971; Peakall and Lovett, 1972; Saha, 1972). Much research has been done on various aspects of mercury in aquatic, animal, and human systems. Extensive data are available on mercury residues in plants resulting from foliar applications (Smart, 1968). Data are sparse, however, on the absorption of mercury into plants from soils.

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Mercury in soil may result from fungicide applications, air pollution, or that present natively. An analytical survey of mercury in 912 samples of soil taken throughout the United States showed levels ranging from 55 to 4600 ppb, with a geometric mean of 71 ppb (Shacklette *et al.*, 1971). From limited data available, it appears that plants rarely contain mercury concentrations above 500 ppb (Shacklette, 1970). Plants may absorb higher concentrations of mercury when grown in proximity to mercury ore deposits or mines but it is possible that mercury in the air in these regions may contribute to their total content (Byrne and Kosta, 1970). It has been reported that jagged chickweed (*Holosteum umbellatum*) and certain algae may concen-