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Fate and Effect of [^{14}C]Fenvalerate in a Tidal Marsh Sediment Ecosystem Model

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The fate and effect of [^{14}C]fenvalerate in a tidal marsh sediment ecosystem model were examined. Two dosages (0.2 and 1.0 ppm) of [^{14}C]fenvalerate were uniformly applied to the sediment. Additionally, a third treatment (1.0 ppm) was applied to the top centimeter of a 3-cm column in order to monitor the vertical movement of the compound (plus metabolites). No adverse effects of [^{14}C]fenvalerate on heterotrophic microorganisms were observed after 7 days at any treatment level, as measured by plate counts and substrate degradation assays (starch, cellulose, and protein). The half-lives of [^{14}C]fenvalerate in the 0.2- and 1.0-ppm noncolumn treatments were 6.3 and 8.9 days, respectively. The lower half-life (3.2 days) observed in the 1.0-ppm column treatment was attributed to a higher leaching rate in that treatment. TLC analysis of sediment extracts revealed the presence of three identifiable metabolites in all treatments after 4 days of incubation.

In the past 25 years, considerable ecological research has been performed concerning energy flow, productivity, and nutrient cycling in salt marsh environments. The high productivity of salt marshes has been attributed to the vertical mixing of water, which produces a nutrient trap that is comparable to land under intensive agriculture (Edwards and Davis, 1975; Pomeroy et al., 1972; Teal, 1962). Microbial degradation of plant material serves as the principal link between primary and secondary production in salt marsh environments. Only small amounts of the predominant marsh grasses (i.e., *Spartina alterniflora*) are consumed while living. Decaying plant material from marsh and terestrial sources provided carbon and energy for the microflora of the marsh sediments (Haines, 1977; Maccubbin and Hodson, 1980). Subsequently, the sediment microflora enrich the nutritive value of the detritus as food for a variety of grazers (Haines and Hansen, 1979; Tenore, 1977). It has recently been determined that heterotrophic bacteria are the most important component in salt marsh sediment in regards to the mineralization process (Fallon and Pfaender, 1976).

Xenobiotics, including many pesticides, enter estuaries as a result of erosion, agricultural runoff, industrial effluents, or by direct application in or near salt marsh environments. The same tidal action that functions as a nutrient trap may cause pesticides to be swept back and forth through the salt marsh at concentrations possibly injurious to productivity. Concern for possible hazards to nontarget species, including disruption of the heterotrophic decomposition process in salt marshes, has prompted studies to determine the fate and effects of these pesticides (Bourquin et al., 1976). In this paper, we report the development and use of a model salt marsh ecosystem to evaluate the fate and effect of a pesticide under simulated salt marsh conditions. [^{14}C]Fenvalerate [cyano(3-phenoxyphenyl)methyl 4-chloro-α-(1-methylethyl)benzeneacetate] was used as the reference pesticide in this study. Fenvalerate is a pyrethroid insecticide that has great potential for control of a wide range of insect pests in agriculture, because it combines outstanding insecticidal activity, moderate mammalian toxicity, and adequate stability in the field. Only a few studies have been performed with the aim of examining the metabolic pathway of fenvalerate in the environment (Ohkawa et al., 1978; Mikami et al., 1980).

MATERIALS AND METHODS

Tidal Marsh Sediment Ecosystem. Eight 10-L capacity glass ecosystem tanks (operating volume = 7 L) were built (Figure 1a). Two equal-sized chambers were formed in each tank by using a glass partition fitted with a capillary drain hole. Sediment containers (six per tank) were placed on a 9-cm glass platform located in one chamber

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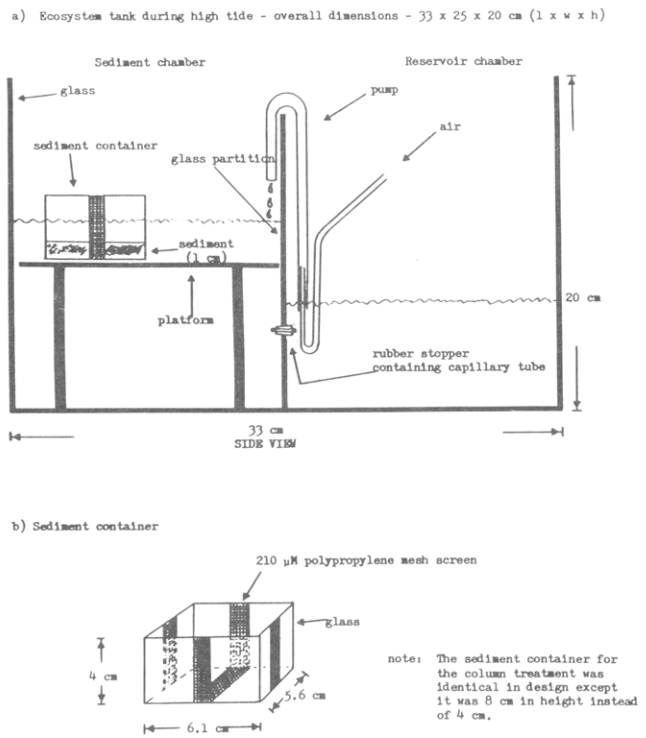

Figure 1. Tidal marsh ecosystem tank and sediment container.

of each tank. The sediment containers were designed to allow chemical movement through the sediment in both lateral and vertical directions. They were constructed by joining 10 pieces of glass (per container) to strips of 210 μM mesh polypropylene screen on all four sides and down the bottom of the container (Figure 1b). All joints (ecosystem tanks and sediment containers) were sealed together with Dow Corning 100% silicone rubber general-purpose sealant. A glass lid containing a small opening (to allow gas exchange) was placed on top of the tank to minimize evaporation and contamination of the system.

Tidal marsh conditions were simulated as follows. Estuarine water (7 L) was placed in the ecosystem tank up to the platform level. Twice every 24 h (11:00 a.m. and 11:00 p.m.), a timer activated air pumps that transferred water from the reservoir side to the platform side of the tank (containing the sediment). After approximately 30 min, the sediment was submerged under 5–6 cm of water, thus representing high-tide conditions. Air pumps were shut off after 3 h. Water, flowing through the partition capillary hole, reached the equilibrium level (platform height) after 45 min, thus representing low-tide conditions. The entire study was run under greenhouse conditions (July 15–Aug 12, 1982; Beltsville, MD).

Sediment/Estuarine Water. The sediment and estuarine water (obtained on July 9 and July 15, 1982, repsectively) were taken from approximately the same location in the Quarter Creek of the Wye River (a tributary of the Chesapeake Bay). The sediment, removed from an area high in *Spartina* spp. growth, was air-dried in the greenhouse for 1 week, sieved at 2 mm, classified as a sand (97.4% sand, 1.1% silt, and 1.5% clay), and contained 0.35% organic carbon. The estuarine water (pH 7.82, salinity 10.2‰) was returned to the laboratory, placed in the ecosystem tanks (7 L/tank), and allowed to acclimate for 24 h under greenhouse conditions.

Test Compound/Sediment Treatment. Nonlabeled fenvalerate, [^{14}C]fenvalerate (total activity = 500 μCi; specific activity = 35.5 μCi/mg; ring labeled in the acid moiety), and fenvalerate standard metabolites were provided by Shell Chemical Co. The radiopurity, as determined by thin-layer chromatography (TLC) and autoradiography, was 97.9%.

Two treatments, designated NC (noncolumn), were prepared by uniformly applying 0.12 mg (0.2 ppm) or 0.6 mg (1.0 ppm) of [^{14}C]fenvalerate (labeled plus unlabeled) to 0.6-kg quantities of sediment (2.7 μCi each rate). Six 50-g portions (dry weight) of the treated sediment were added to sediment containers and placed in duplicate ecosystem tanks. A third treatment, designated CM (column), was prepared in order to monitor the vertical movement of [^{14}C]fenvalerate. Twelve columns (3 cm deep) were prepared by separating 50-g (1-cm thick) quantities of sediment with 210 μM mesh screen. Wires attached to the corners of each screen facilitated the removal of each layer (top, middle, and bottom) of sediment without contaminating the adjacent layers. The top layer of sediment (in each of the 12 columns) was uniformly treated with 0.15 mg of [^{14}C]fenvalerate (3.0 ppm; 0.68 μCi). (This treatment is refered to as the 1.0-ppm column treatment). The sediment containers were then immediately placed in duplicate ecosystem tanks. The control treatment consisted of sediment treated with an amount of solvent (ethyl ether) equivalent to that used in the high-dose treatment (0.055 mL/g or 33 mL/600 g of sediment).

Sampling. Immediately following treatment, one sediment container (50 g of sediment) was removed from each ecosystem tank (day 0 sample) for analysis. Additional 50-g sediment samples were removed at 1, 4, 7, 14, and 28 days for microbial and chemical analysis plus soil moisture determinations. At each sampling, the water in each tank was analyzed for pH, temperature, dissolved oxygen, salinity, and general appearance.

Microbiology. Heterotrophic bacteria were enumerated by using standard plate-count techniques (American Public Health Association, 1975). Serial dilutions to 10^{-5} were prepared from 1.0 g of sediment (dry weight) and spread-plated in duplicate on Marine Agar (Difco). Plates were incubated at 30 °C for 5 days, and colony forming units (CFU) were counted on plates within the statistically valid range (30–300 CFU/plate). Plate count reproducibility was ±10%.

Long-term effects of fenvalerate on microbial degradation of starch, cellulose, and protein was monitored by an in vitro radiometric technique (Buddemeyer, 1974; Buddemeyer et al., 1976; Caplan et al., 1981; Isbister et al., 1980). After 28 days, duplicate sediment aliquots (0.5 g) from each chamber were evenly mixed with 0.1 μCi of water-soluble U-^{14}C tobacco starch (catalog no. 11134, ICN Chemical Co.), 0.1 μCi of U-^{14}C-cellulose (*Canna indica*; catalog no. 11007, ICN Chemical Co.), or 0.1 μCi of water-soluble ^{14}C algal protein (*Chlorella sorokiniana*; catalog no. 10032, ICN Chemical Co.). Each sediment sample was placed in a sterile 0.5-dram vial that was subsequently placed inside a 20-mL liquid scintillation vial containing a filter strip (Whatman No. 1) saturated with concentrated scintillation cocktail. The vials were immediately sealed and cumulative $^{14}CO_2$ evolution was monitored over a 24-h period. Total cpm from the treated sediment was compared to that of the control (and reported as percent of control).

Combustion and Liquid Scintillation Counting (LSC). Sediment samples (250 mg) were combusted (Packard Model 306 Tri-Carb sample oxidizer) in duplicate (corrected for combustion efficiencies of 94%) and counted in Permafluor liquid scintillation cocktail (Packard). Water samples from each chamber were counted directly

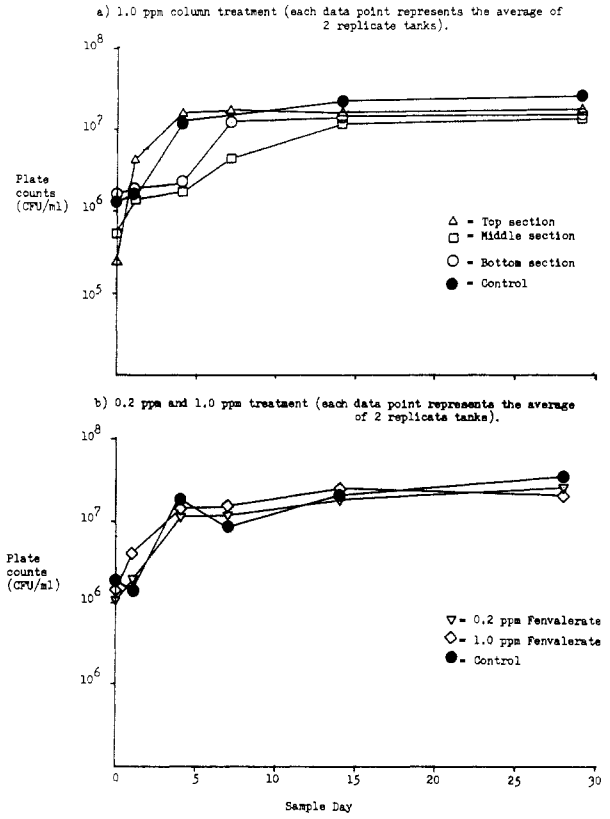

Figure 2. Effect of fenvalerate on heterotrophic bacterial populations in tidal marsh sediment (grown on Difco Marine Agar).

by addition of 1.0 mL of water to HP Ready-Solv liquid scintillation cocktail (Beckman). All radioassay quantitations were performed using a Model LS-6800 liquid scintillation spectrometer (using H# for quench calculations).

Extraction and Thin-Layer Chromatography. Sediment samples (20 g) were Soxhlet extracted for 16 h with 150 mL of methanol. Aliquots (1.0 mL) from the extracts were removed and counted for radioactivity to determine the efficiency of extraction. The extracts were then dried through sodium sulfate and evaporated to near dryness under a stream of nitrogen. Water samples (200 mL) from the 28-day column-treated chambers were acidified and partitioned with ethyl acetate (2 × 200 mL). The combined ethyl acetate fractions were dried through sodium sulfate and evaporated to near dryness under a stream of nitrogen.

The water and sediment extracts were adjusted to approximately 5000–6000 dpm in 50 μL for spotting on Kieselgel 60 precoated silica gel TLC plates (0.25-mm thickness, fluorescent indicator). The extracts were spotted alongside the fenvalerate standard metabolites (as well as overspotted with 5 μg of nonlabeled fenvalerate standard) and developed in two different solvent systems—hexane–acetone–acetic acid (25:25:1, solvent system 1) and toluene–ether–acetic acid (75:25:1, solvent system 2). Radioactive compounds on the TLC plates were located by autoradiography, using Kodak-XAR X-ray film.

RESULTS AND DISCUSSION

The tidal marsh ecosystems performed without incident throughout the experimental period. Very little algae fouling occurred in any of the tanks, and the caplillary tube (permitting water flow from the sediment chamber to the reservoir chamber) only had to be cleared once. Due to the presence of the tank lids, water loss was minimal (≃7%). The ecosystems were therefore relatively maintenance free. No appreciable differences (±5%) in the physical measurements (salinity, dissolved oxygen, pH, and temperature) of the tank water occurred in any of the treatments. As expected, water temperature fluctuated with that of the external environment (i.e., greenhouse), but little variation occurred between tanks (±0.5 °C). Dissolved oxygen (7.7 ± 0.5 mg/L) and pH (8.50 ± 0.50 pH units) remained constant for all tanks throughout the study.

Table I. Degradation of Starch, Cellulose, and Protein 28 Days following the Application of [^{14}C]Fenvalerate to Tidal Marsh Sediment[a]

[^{14}]fenvalerate treatment	degradation[b] starch	cellulose	protein
0.2 ppm (tanks 5 and 6)	104.3	101.1	93.3
1.0 ppm (tanks 3 and 4)	110.9	85.6	82.4
1.0 ppm (tanks 1 and 2)			
top section	103.9	104.0	97.2
middle section	103.6	126.7	99.9
bottom section	111.1	107.9	102.2

[a] Duplicate sediment samples were analyzed from each tank and averaged. The values from the duplicate tanks (i.e., 1 and 2, 3 and 4, and 5 and 6) were then averaged together, providing a total of four replicates per treatment. The average error = ±9%. [b] Degradation values are based on a 24-h incubation period and are reported as percent of control (7059 cpm for starch control; 3080 cpm for cellulose control; 12 872 cpm for protein control).

The effect of [^{14}C]fenvalerate on heterotrophic bacteria in the sediment is shown in Figure 2. Throughout the study, no appreciable differences (±10%) in plate counts were observed between both noncolumn (NC) and sediment treatments (0.2 and 1.0 ppm) of [^{14}C]fenvalerate and those of the control (Figure 2b). Plate counts for both control and [^{14}C]fenvalerate NC treated sediments averaged 1.7 × 10^6 CFU/mL on day 0 and steadily increased to 3.8 × 10^7 CFU/mL by day 28. However, some initial differences in plate counts were found between the 1.0-ppm column (CM) treated sediment and that of the control sediment (Figure 2a). On day 0, plate counts were 4.2 × 10^5 CFU/mL in the top section of sediment column compared with 2.0 × 10^6 CFU/mL in the control sediment. This difference cannot be explained without further toxicological tests. After 4 days, bacterial levels were back up to control levels (coinciding with a decrease in fenvalerate concentration) and remained so for the remainder of the study. All sediments were represented by a heterogeneous bacterial population throughout the experiment.

The in vitro radiometric technique revealed that, after 28 days, [^{14}C]fenvalerate did not affect the ability of sediment to degrade ^{14}C-starch, ^{14}C-cellulose, or ^{14}C-protein at any of the treatment levels tested (Table I). In most cases, substrate degradation in the treated sediment was equal to (or greater than) substrate degradation in the control sediment. However, it must be noted that this was only one sampling and does not necessarily reflect any early degradation patterns of the sediment.

Radiocarbon recovery averaged 96.3% of the applied dose in the NC treated sediments (Table II). Initial (day 0) sediment radiocarbon levels were ≃100% and steadily decreased to an average of 69.4% by day 28. Most of the remaining radiocarbon was found in the tank water, averaging 22.3% and 28.1% of the applied radiocarbon in day 28 for the 0.2- and 1.0-ppm [^{14}C]fenvalerate treatments, respectively. The leaching of compound from the sediment is not surprising since the sediment contained such a high sand content (97.4%). The leaching of radioactivity was more dramatically shown in the CM treated sediment (Table III). After just 1 day of incubation, only

Table II. Recovery of Radiocarbon following the Addition of 0.2 or 1.0 ppm of [14C]Fenvalerate to a Tidal Marsh Sediment Ecosystem (NC Treatment)

treatment, ppm	tank no.	day	recovery of radiocarbon,[a] ppm: sediment	water	total recovery
0.2	5 (rep. 1)	0	106.9 (0.21)		106.9
		1	103.3 (0.21)	b	103.3
		4	92.1 (0.18)	2.4	94.5
		7	91.2 (0.18)	1.3	92.5
		14	84.5 (0.17)	6.5	91.0
		28	71.1 (0.14)	25.6	96.7
					$\bar{X}$ = 97.5
	6 (rep. 2)	0	106.6 (0.21)		106.6
		1	97.5 (0.20)	2.2	97.7
		4	96.1 (0.19)	1.4	97.5
		7	97.8 (0.20)	<0.1	97.8
		14	86.0 (0.17)	8.7	94.7
		28	66.0 (0.13)	18.9	84.9
					$\bar{X}$ = 96.5
1.0	3 (rep. 1)	0	103.8 (1.04)		103.8
		1	96.5 (0.97)	3.6	101.1
		4	92.1 (0.92)	<0.1	92.1
		7	91.2 (0.91)	9.5	91.7
		14	76.9 (0.77)	9.7	86.6
		28	69.0 (0.69)	24.4	93.4
					$\bar{X}$ = 94.8
	4 (rep. 2)	0	100.3 (1.00)		100.3
		1	97.2 (0.97)	b	97.2
		4	88.7 (0.89)	5.5	94.2
		7	96.8 (0.97)	<0.1	96.8
		14	83.2 (0.83)	3.3	86.5
		28	71.5 (0.72)	31.8	103.3
					$\bar{X}$ = 96.4

[a] Values given in percent of dose. [b] Values too low to statistically quantitate.

64.6% of the applied radiocarbon remained in the top 1-cm section of the sediment column (compared with >96% radioactivity in the NC treated sediment). The balance of radiocarbon was accounted for in the middle (18.2%) and bottom (6.1%) sediment sections plus the water (4.0%). However, the radiocarbon levels remained relatively constant in each sediment section (with the exception of the day 28 tank 1 sample) for the rest of the experiment. Approximately 13% of the applied radioactivity was observed in the tank water by day 28. Overall radiocarbon recoveries in the CM treated sediment average 87.5% of the applied dose.

Since fenvalerate and many of its metabolites are insoluble in water, it is not surprising that the compound (plus metabolites) moved vertically down the sediment column rather than laterally into the water phase. Additional factors involved in the movement of a compound through sediment include the intensity and frequency of water flow, sediment properties (i.e., pH, cation-exchange capacity, anion-exchange capacity, percent organic matter, and particle size), and the compound properties (Weber and Peeper, 1977). The organic carbon content of the sediment was lower than expected (0.35%). As a result, vertical movement was probably not as greatly influenced by organic carbon as it would be in sediments containing higher levels of organic carbon. Another possible explanation is that the top 1-cm section of the CM sediment became saturated with compound. (The top 1-cm sediment section actually contained 3.03 ppm of fenvalerate on day 0 compared with fenalverate concentrations of 0.2 and 1.0 ppm in the NC treatments). The compound then precipitated out and moved into the lower sediment sections upon flooded conditions. This may also explain the lower overall radioacarbon recoveries (87.5%) observed in

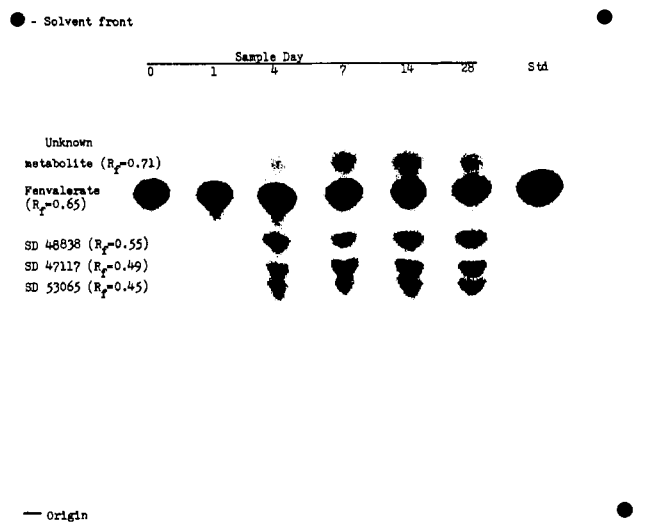

Figure 3. TLC autoradiogram of tidal marsh sediment extracts, following treatment wtih [14C]fenvalerate to sediment (TLC solvent system 1).

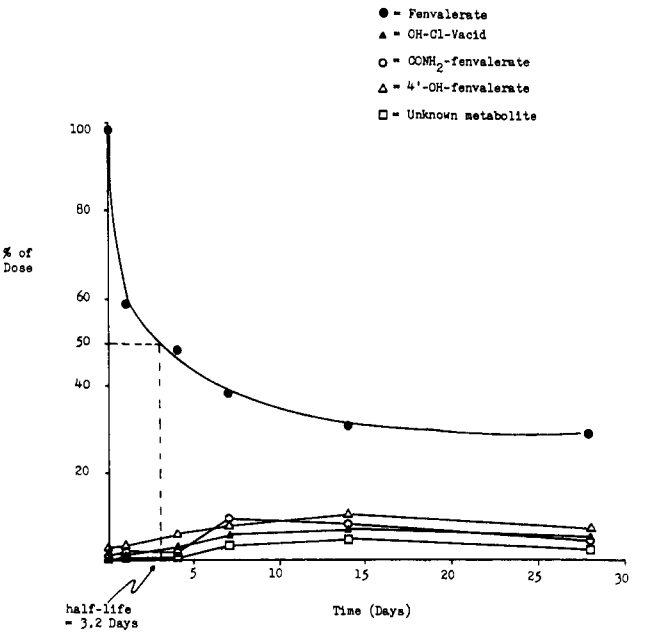

Figure 4. Disappearance of fenvalerate and appearance of metabolites in tidal marsh sediment treated with 1.0 ppm of [14C]fenvalerate (top section of the column).

the CM treated sediments. (However, it should be noted that the glass was not analyzed for possible bound 14C material.)

The extraction of radiocarbon from sediment averaged 95.7% for all treatments. There was no consistent upward trend in bound residues at any treatment throughout the study. The qualitative degradation pathway was similar in all [14C]fenvalerate treatments. Thin-layer chromatographic analysis (solvent system 1) of the sediment extracts revealed the presence of four metabolites in all samples after day 4, as shown in Figure 3. Cochromatographic comparison with standard metabolites revealed the identity of three of these metabolites: OH-Cl-Vacid (SD 53065), R_f = 0.45; $CONH_2$-fenvalerate (SD 47117), R_f = 0.49; 4'-OH-fenvalerate (SD 48838), R_f = 0.55. The identity of these metabolites was confirmed by TLC solvent system 2. None of the R_f values of the standard metabolites provided by Shell Chemical Co. corresponded to that of the fourth metabolite (R_f = 0.71).

Radiocarbon recoveries of the scraped TLC plates of the sediment extracts are shown in Figures 4–6. The inter-

Table III. Recovery of Radiocarbon following the Addition of 1.0 ppm of [^{14}C]Fenvalerate (Column Treatment) to a Tidal Marsh Sediment Ecosystem

		recovery of radiocarbon [a] ppm				
		sediment[b]			water	
tank no.	day	top	middle	bottom	(chamber)	total ^{14}C
1 (rep. 1)	0	101.1 (3.03)	5.9 (0.18)	1.9 (0.06)		108.9
	1	62.2 (1.87)	19.8 (0.59)	6.3 (0.19)	3.7	92.0
	4	69.6 (2.09)	6.3 (0.19)	10.3 (0.31)	5.8	92.0
	7	67.3 (2.02)	6.7 (0.20)	6.4 (0.19)	<0.1	80.4
	14	58.5 (1.76)	10.9 (0.33)	5.7 (0.17)	<0.1	75.1
	28	42.4 (1.27)	10.9 (0.33)	5.5 (0.17)	17.1	75.9
						$\bar{X}$ = 87.4
2 (rep. 2)	0	[c]	[c]	[c]	[c]	[c]
	1	67.0 (2.01)	16.6 (0.50)	5.8 (0.17)	4.2	89.4
	4	58.9 (1.77)	17.5 (0.53)	11.4 (0.34)	<0.1	87.8
	7	64.3 (1.93)	17.0 (0.51)	13.9 (0.42)	<0.1	95.2
	14	65.4 (1.96)	10.9 (0.33)	3.0 (0.09)	6.8	86.1
	28	58.3 (1.75)	6.9 (0.21)	4.5 (0.14)	9.2	78.9
						$\bar{X}$ = 87.5

[a] Values given in percent of dose. [b] Sediment columns were divided into three 1-cm (50-g) sections. [c] Sample lost.

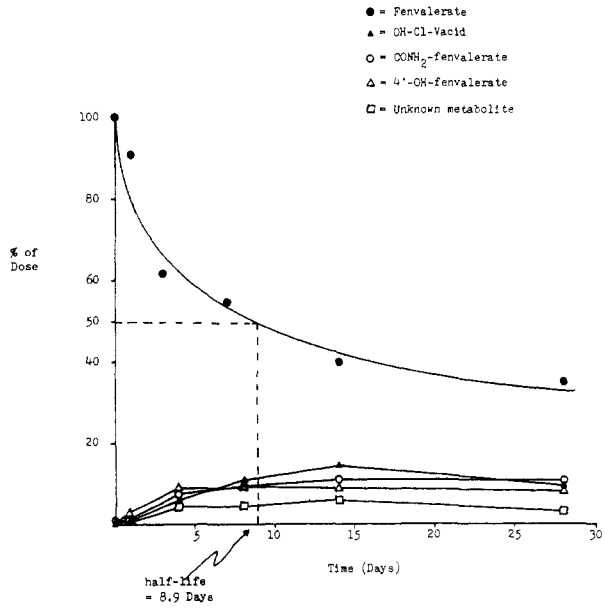

Figure 5. Disappearance of fenvalerate and appearance of metabolites in tidal marsh sediment treated with 1.0 ppm of [^{14}C]fenvalerate (noncolumn treatment).

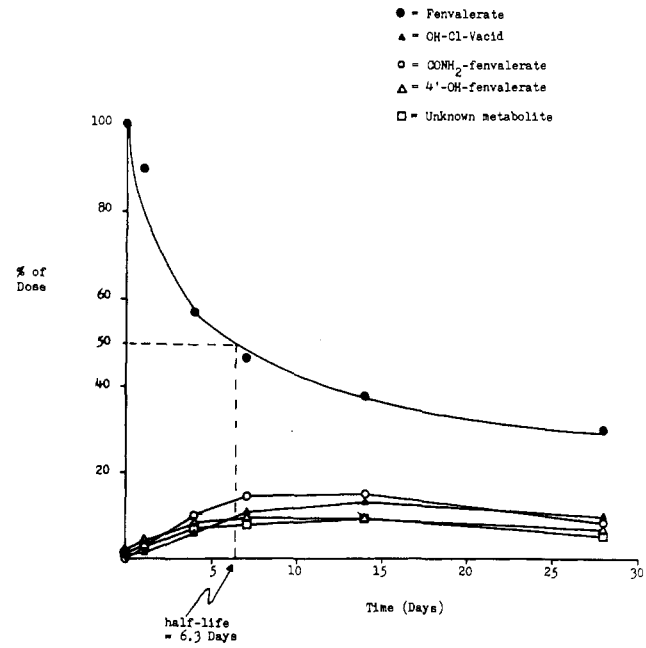

Figure 6. Disappearance of fenvalerate and appearance of metabolites in tidal marsh sediment treated with 0.2 ppm of [^{14}C]fenvalerate (noncolumn treatment).

polated half-lives of fenvalerate in the 1.0-ppm CM (top section), 1.0-ppm NC, and 0.2-ppm NC treatments were 3.2, 8.9, and 6.3 days, respectively. Since more leaching occurred in the CM treated sediment (on day 0), it is not surprising that the half-life of fenvalerate was much lower than in the NC treated sediment. Approximately 31% of the applied radiocarbon was observed as fenvalerate in all sediments by day 28. The four metabolites accounted for 20% of the radioactivity in the CM treated sediment on day 28 (Figure 4) compared with a total of 34% in the NC treatments (Figures 5 and 6).

TLC development (solvent system 2) of the day 28 aqueous extracts (tanks 1 and 2 combined) from the 1.0-ppm CM treatment revealed the presence of only one product, Cl-Vacid (SD 44064).

The proposed degradation pathway for [^{14}C]fenvalerate in tidal marsh sediment is shown in Figure 7. Fenvalerate can undergo either a ring hydroxylation to produce 4'-OH-fenvalerate or a hydration at the CN group to produce $CONH_2$-fenvalerate. These products may subsequently undergo ester cleavage to form the products Cl-Vacid (in water) or OH-Cl-Vacid (in sediment) plus a variety of potential unidentifiable metabolites (unlabeled) at the alcohol end of the molecule. Fenvalerate may also undergo direct ester cleavage to form those metabolites. (It should be noted that this pathway is purely speculative since the degradation of individual metabolites was not examined.)

This scheme is consistent with the degradation pathway of fenvalerate in soils as determined by Ohkawa et al. (1978). They found that under aerobic conditions, fenvalerate can undergo the ring hydroxylation reaction as well as the hydration reaction. Again, ester cleavage of both products resulted in Cl-Vacid followed by a decarboxylation reaction, resulting in the release of CO_2. (Since the compound used in our study was not labeled at the carboxyl moiety, this decarboxylation reaction was not included in Figure 7). Under anaerobic conditions, they found that fenvalerate breakdown was initiated by hydration at the CN group to form $CONH_2$-fenvalerate. $CONH_2$-fenvalerate was then degraded to Cl-Vacid as previously described. Fenvalerate can also be degraded by photolytic action as shown by Mikami et al. (1980). Upon exposure to sunlight, hydration at the CN group of

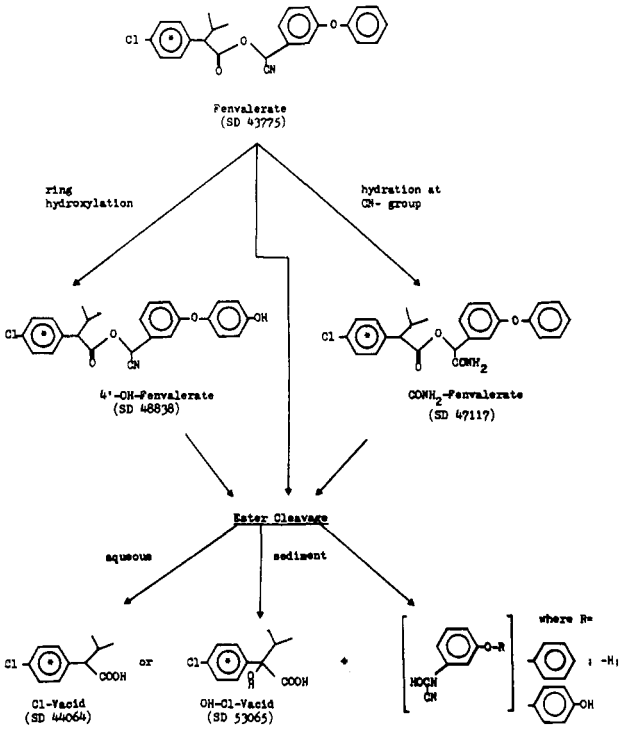

Figure 7. Proposed degradation pathway for fenvalerate in tidal marsh sediment.

fenvalerate (to form $CONH_2$-fenvalerate) was the predominant reaction on soil surfaces. Additional photodegradation reactions occurred via oxidation, decarboxylation, cleavage of ester or diphenyl ether linkages, and hydrolysis from $CONH_2$-fenvalerate to COOH-fenvalerate.

It is impossible to determine the exact mechansim(s) (i.e., microbial, hydrolytic, and photolytic) responsible for degrading fenvalerate in our study since no sterile or dark controls were run. However, it seems likely that fenvalerate was primarily degraded via microbial metabolism and/or hydrolysis rather than by photolysis. The reason for this is that the compound was uniformly applied to the sediment at the initiation of the study; therefore, only a small portion of the compound was exposed to light (via surface).

In this study, fairly good reproducibility (±10%) among replicate ecosystem tanks was shown in regards to physical measurements, plate counts, radiocarbon recovery, and quantitative and qualitative degradation patterns.

Registry No. Fenvalerate, 51630-58-1; OH-Cl-Vacid, 88036-36-6; $CONH_2$-fenvalerate, 67685-93-2; 4′-OH-fenvalerate, 67882-25-1.

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COMMUNICATIONS

Determination of Allidochlor Residues in Pre- and/or Postemergence-Treated Leeks

An analytical method for the gas chromatographic determination of the herbicide allidochlor (2-chloro-*N*,*N*-diallylacetamide) in leeks (*Allium porrum* L.) has been developed using a nitrogen-specific flame ionization detector. The limit of detection of the analytical method was 100 ppb based on a 5 g fresh-weight equivalent, with recoveries being in the order of 75% at the 100-ppb fortification level. Allidochlor residues in mature leeks were less than 100 ppb following either a 7.0 kg/ha preemergence application or a 7.0 kg/ha preemergence plus one or two 7.0 kg/ha postemergence applications.

Currently, allidochlor (2-chloro-*N*,*N*-diallylacetamide) is solely registered in Canada for use on onions with both preemergence and/or postemergence applications at 6.75 kg/ha being used to control annual grasses and broadleaved weeds. Leeks, an onion-like crop, are grown on a limited hectarage in Eastern Canada, with the major

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