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Pyrethroid Insecticides Derived from [1,1′-Biphenyl]-3-methanol

Ernest L. Plummer* and David S. Pincus

The relationship of changes in southern armyworm (*Spodoptera eridania*) topical activity to variation of the physical and chemical properties of meta substituents was examined for a series of meta-monosubstituted benzyl esters of *cis,trans*-3-(2,2-dichloroethenyl)-2,2-dimethylcyclopropanecarboxylic acid. With the exception of substituents containing two-atom bridges between the benzyl ring and a second aromatic ring, there was a significant dependence of the activity on the oil/water partitioning property (π) of the substituent. The phenyl substituent fits this relationship closely, indicating that it is not necessary to have a bridging group between the aromatic rings of 3-substituted benzyl alcohols in order for their esters to display high pyrethroid-like activity. ([1,1′-Biphenyl]-3-yl)methyl *cis,trans*-3-(2,2-dichloroethenyl)-2,2-dimethylcyclopropanecarboxylate was found to have initial and residual activity paralleling that of permethrin against a number of insects.

The alcohol portion of most active pyrethroids contains two centers of unsaturation separated by a bridging atom. In allethrin and resmethrin this structural feature is represented by the carbon atom of the methylene groups while in permethrin the bridging atom is oxygen. Qualitative discussions of structure–activity relationships of pyrethroids have generally pointed to this feature as a requirement for good insecticidal activity. At one time it was suggested that the bridging group may actually perform a function at the active site (Elliott, 1969), while more recently it has been suggested that the lack of coplanarity between the centers of unsaturation, that results from the presence of the bridging group, provides optimum fit at the active site (Elliott et al., 1974).

We wish to report a quantitative study of the structure–activity relationships of meta-monosubstituted benzyl esters of *cis,trans*-3-(2,2-dichloroethenyl)-2,2-dimethylcyclopropanecarboxylic acid (DVA) conducted with the intent of either expressing the requirements for this bridging group quantitatively or finding alternate requirements for activity for such esters. Additionally, we wish to report a new series of insecticidal pyrethroid esters of [1,1′-biphenyl]-3-methanol that were prepared as part of this study.

MATERIALS AND METHODS

Chemicals. Esters and α-cyano esters of all pyrethroid acids cited were prepared by one of the three methods described below. Acceptable elemental and spectroscopic data were obtained for all novel compounds.

([1,1′-Biphenyl]-3-yl)methyl cis,trans-3-(2,2-Dichloroethenyl)-2,2-dimethylcyclopropanecarboxylate. (1) Method A. A solution of [1,1′-biphenyl]-3-methanol (0.025 mol), prepared by the method of Hammond and Reeder (1958), DVA chloride (cis/trans = 40:60) (0.025 mol), and pyridine (0.025 mol) in methylene chloride was stirred at room temperature for 16 h. The mixture was taken up in water and extracted with chloroform. The chloroform extracts were washed sequentially with 2 N HCl, saturated salt solution, and 2 N NaOH. The dried solution ($MgSO_4$) was concentrated and short path distilled at 130 °C/0.25 mmHg to give an 82% yield of oily product: NMR ($CDCl_3$) δ 1.15 (s), 1.22 (s), 1.26 (s), 1.30 (s), 1.62–2.41 (m), 5.19 (s), 5.63 (d), 6.34 (dd), 7.20–7.77 (m). Anal. Calcd for $C_{21}H_{20}Cl_2O_2$: C, 67.21; H, 5.37. Found: C, 67.39; H, 5.66.

(2) *Method B.* To an aqueous solution of potassium hydroxide (0.032 mol) was added *cis,trans*-DVA (cis/trans = 40:60) (0.032 mol). When all the acid had dissolved, 100 mL of heptane was added and the water removed by distillation. The dried mixture was cooled to 60 °C, and an acetonitrile solution of 3-(bromomethyl)-1,1′-biphenyl (0.032 mol), prepared by the method of Grovenstein and Wentworth (1967), and 0.1 g of 1,4-diazobicyclo[2.2.2]octane was added. The mixture was refluxed for 5.5 h and worked up by a procedure similar to that in method A.

([1,1′-Biphenyl]-3-yl)cyanomethyl cis,trans-3-(2,2-Dichloroethenyl)-2,2-dimethylcyclopropanecarboxylate. A tetrahydrofuran–water solution of the cyanohydrin of [1,1′-biphenyl]-3-carboxaldehyde (0.013 mol), which was prepared from 3-(bromomethyl)-1,1′-biphenyl by the Sommelet procedure, was combined with *cis,trans*-DVA chloride. The mixture was stirred for 16 h at room temperature and then diluted with water. The organic layer, combined with a chloroform extract of the aqueous layer, was washed sequentially with saturated $NaHCO_3$, saturated NaCl, saturated $Na_2S_2O_3$, and saturated NaCl solutions and dried ($MgSO_4$), and the solvent was removed. Silica gel chromatography (20% hexane–$CHCl_3$) gave 3.3 g or 63% of oily product: NMR ($CDCl_3$) δ 1.17 (s), 1.22 (s), 1.32 (s), 1.35 (s), 1.65–2.45 (m), 5.63 (dd), 6.23 (d), 6.47 (s), 6.50 (s), 7.27–7.77 (m) (cis/trans = 36:64). Anal. Calcd for $C_{22}H_{19}Cl_2NO_2$: C, 66.01; H, 4.78. Found: C, 66.41; H, 5.22.

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Table I. Substituent Parameters for Meta-Monosubstituted Benzyl Esters of *cis,trans*-3-(2,2-Dichloroethenyl)-2,2-dimethylcarboxylic Acid

no.	substituent	log [1/(*RP*)]	π	σ_m	F	R	M_r	L	B_1	B_4
1	OC_6H_5[a]	0.00	2.08	0.25	0.33	−0.12	27.68	4.51	1.35	5.89
2	C_6H_5	0.25	1.96	0.06	0.08	−0.03	25.36	6.28	1.70	3.11
3	$CH_2C_6H_5$[a]	0.45	2.01	−0.08	−0.08	0.00	30.01	3.63	1.52	6.02
4	I	1.54	1.12	0.35	0.39	0.07	13.94	4.23	2.15	2.15
5	$OCH_2C_6H_5$	1.77	1.66				32.19	8.20	1.35	3.11
6	CF_3	1.85	0.88	0.43	0.37	0.07	5.02	3.30	1.98	2.61
7	$CH_2CH_2C_6H_5$	1.91	2.66				34.65	8.33	1.52	3.15
8	$CH{=}CHC_6H_5$	2.03	2.68	0.03	0.06	−0.04	34.17			
9	H[b]	2.17	0.00	0.00	0.00	0.00	1.03	2.06	1.00	1.00
10	Br	2.17	0.86	0.39	0.43	−0.06	8.88	3.83	1.95	1.95
11	Cl	2.17	0.71	0.37	0.40	−0.50	6.03	3.52	1.80	1.80
12	NO_2	2.28	−0.28	0.71	0.67	0.16	7.36	3.44	1.70	2.44
13	OCH_3	2.32	−0.02	0.12	0.25	−0.18	7.87	3.98	1.35	2.87
14	CH_3	2.34	0.56	−0.07	−0.04	−0.05	5.65	3.00	1.52	2.04
15	F	2.42	0.14	0.34	0.42	−0.12	0.92	2.65	1.35	1.35
16	CO_2CH_3	2.59	−0.01	0.37	0.32	0.05	12.87	4.85	1.90	3.36
17	$C{\equiv}CC_6H_5$	3.40	2.65	0.14	0.12	0.02	33.21	8.88	1.70	3.11
18	$NHCOCH_3$	inactive	−0.97	0.21	0.27	−0.08	14.93	5.15	1.50	3.61
19	$CH(CH_3)_2$	inactive	1.53	−0.07	−0.05	−0.03	14.98	4.11	2.04	3.16

[a] Compounds previously reported by Elliott et al. (1975). [b] Compound previously reported by Kondo et al. (1977).

Biological Studies. Southern armyworm, *Spodoptera eridania*, was from a laboratory-reared culture maintained on a modified pinto bean diet (Shorey and Hale, 1965).

In the topical tests, toxicants were applied in acetone (0.1–1.0 μL), by using an Arnold microapplicator, to the second dorsal thoracic segment of third stadia larvae. The larvae were incubated at room temperature in 9-cm Petri dishes lined with moist filter paper and containing a piece of pinto bean diet. So that a dosage mortality line could be established, at least five dosages using two replications of 10 insects per treatment were tested. Mortality was assessed after 24 h at room temperature.

In foliar tests, bush bean var. Pinto (*Phaseolus vulgaris*) at the bifoliate leaf stage was sprayed to runoff with the experimental compound suspended in acetone–water (10:90). After the plants dried, the treated leaves were excised, placed in a 16-oz paper cup, and infested with 10 second stadia larvae. The cups were capped and incubated at room temperature for 48 h after which time insect mortality was assessed.

Relative potency of each compound was calculated as the ratio of the LD_{50} of permethrin (included in all tests as the standard) to that of the compound.

Other insects used were cabbage looper (*Tricholpusia ni*), tobacco budworm (*Heliothis virescens*), Mexican bean beetle (*Epilachna varivestis*), large milkweed bug (*Oncopeltus fasciatus*), and pea aphid (*Acyrthosiphon pisum*).

Structure–Activity Studies. Values of π, σ_m, F, R, and M_r published by Hansch et al. (1973a) were used for structure–activity correlations. Values of π were used without regard to position. The values of π for 11 of the 19 substituents (1, 2, 5–7, and 10–15) were correlated with log P values determined experimentally by an HPLC method similar to that used by McCall (1975). Excellent correlation was found (r = 0.950; s = 3.60; F = 82). F and R values were scaled to the meta position by using the scaling factors published by Noorington et al. (1975). The steric factors L, B_1, and B_4 were derived from the tabulations published by Verloop et al. (1976). The values of these parameters used for multiple linear regression analysis are shown in Table I.

Many of the substituents were chosen either because the required alcohols were available commercially or because they were part of ongoing studies in our laboratory. However, an attempt was made to cover the maximum parameter space by application of cluster analysis. The 20 cluster level of set 1 of the cluster sets published by Hansch et al. (1973b) was used to select additional substituents.

Two substituents, the benzyl and the phenyl group, were chosen from the same cluster as the phenoxy substituent. The phenyl substituent was chosen to represent an aromatic ring that lacked the bridging atom in question.

RESULTS AND DISCUSSION

Seventeen of the nineteen meta-monosubstituted (see Table I for substituents) benzyl esters of *cis,trans*-3-(2,2-dichloroethenyl)-2,2-dimethylcyclopropanecarboxylic acid prepared for this study were active (LD_{50} < 20 μg) against southern armyworms (*S. eridania*). When the data from all the active compounds were analyzed by multiple linear regression, it was found that the greatest amount of variance could be explained by equations generated by using the Verloop steric factors.

An equation explaining 46% of the variance in southern armyworm response was generated by using the length (L) and maximum radius (B_4) parameters (eq 1). Since a

$$\log [1/(RP)] = 0.11L - 0.47B_4 + 2.68 \quad (1)$$

$$n = 16 \quad r = 0.690 \quad s = 0.702 \quad F = 5.9$$

negative coefficient leads to an increase in activity, this equation indicates that an increase in maximum radius will increase activity while an increase in the length will decrease activity.

The bridged substituents are unsymmetrical, with larger widths than lengths (i.e., L/B_4 < 1.00). Therefore, a dependence of the activity on the L/B_4 ratio could be better evidence than L or B_4 that a bridging group was required. However, considerably less of the variance is explained (eq 2) by the use of the ratio of length to maximum radius.

$$\log [1/(RP)] = 0.71L/B_4 + 0.60 \quad (2)$$

$$n = 16 \quad r = 0.499 \quad s = 0.811 \quad F = 4.6$$

Unlike L and B_4, the ratio, L/B_4, is not cross correlated with π, and eq 3 was generated, explaining 56% of the

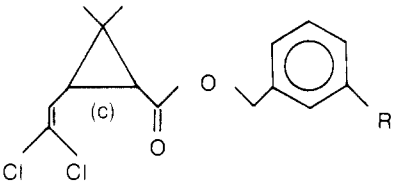
$$\log [1/(RP)] = 0.94L/B_4 + 0.53\pi + 0.75 \quad (3)$$

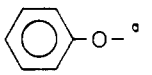
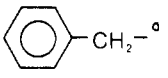
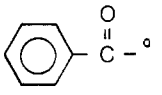
$$n = 16 \quad r = 0.747 \quad s = 0.645 \quad F = 8.2$$

biological variance.

Since at best less than 60% of the variance could be explained by either the L/B_4 ratio or the Verloop parameters, in general it was concluded that little evidence ex-

Table II. Insecticidal Activity of 3-Aryl-Substituted Benzyl Esters



R	Topical Relative Potency					Foliar Relative Potency	
	SAW	CL[b]	TBW[c]	MBB[d]	MWB[e]	SAW	PA[f]
 (π = 2.08)	2.4	1.1	1.8	2.0	2.7	1.1	1.4
 (π = 1.96)	1.1	0.3	0.4	1.0	1.3	0.6	1.3
 (π = 2.01)	0.4	0.3	0.5	0.3	0.8		0.8
 (π = 1.05)	0.4	0.5	0.5	0.3	0.6	0.1	0.5

[a] Compound originally reported by Elliott et al. (1975). [b] Cabbage looper (*T. ni*). [c] Tobacco budworm (*H. virescens*). [d] Mexican bean beetle (*E. varivestis*). [e] Large milkweed bug (*O. fasciatus*). [f] Pea aphid (*A. pisum*).

isted for the requirement of a bridging atom to assure fit at the active site.

When the southern armyworm response is graphed against the hydrophobic substituent constant (π) (Figure 1), it is apparent that four substituents (substituents **5**, **7**, **8**, and **17**) contribute heavily to the lack of correlation of π with activity. These four substituents have a feature in common that is not present in the other substituents; that is, they possess an aromatic ring separated from the benzyl ring by a two-atom bridge. On the basis of this common feature, these substituents were considered outliers and were dropped from the analysis. Although other parameters still gave poor correlations with activity, π and M_r, individually, were now found to explain most of the variance in southern armyworm response (eq 4 and 5). Since

$$\log [1/(RP)] = -0.08M_r + 2.70 \quad (4)$$

$$n = 13 \quad r = 0.900 \quad s = 0.409 \quad F = 46.8$$

$$\log [1/(RP)] = -1.01\pi + 2.51 \quad (5)$$

$$n = 13 \quad r = 0.929 \quad s = 0.348 \quad F = 68.7$$

M_r and π are heavily cross correlated ($r = 0.848$), it is not surprising that their correlations with activity are similar. The solid line in Figure 1 was generated from eq 5.

As indicated by the broken line in Figure 1, there appears to be eight substituents (**1**, **2**, **3**, **4**, **6**, **10**, **11**, and **14**) that make up a linear portion of this curve. Equation 6 represents this line.

$$\log [1/(RP)] = -1.49\pi + 3.25 \quad (6)$$

$$n = 8 \quad r = 0.990 \quad s = 0.145 \quad F = 302.7$$

We have prepared *cis*-DVA esters of seven of the eight substituents in this linear portion of the plot in Figure 1 (the cis isomer of compound **11** was unavailable). In addition, the *cis*-DVA ester of (3-benzoylphenyl)methanol was prepared and tested. Equation 7 indicates that the

$$\log [1/(RP)] = -1.43\pi + 2.93 \quad (7)$$

$$n = 8 \quad r = 0.877 \quad s = 0.515 \quad F = 20.0$$

cis esters also show a good correlation with π.

A comparison of the ([1,1′-biphenyl]-3-yl)-, (3-phenoxyphenyl)-, (3-benzoylphenyl)-, and [3-(phenylmethyl)-phenyl]methyl esters of *cis*-DVA against a number of species in both topical and foliar evaluations (see Table II) indicates a general compliance with the trends already described for southern armyworms.

The activity of all compounds is clearly not fully described by π alone. The benzoyl-substituted compound is generally more active than predicted by π alone while the phenylmethyl compound is less active. Since, in addition, the isopropyl-substituted ester (substituent **19**, Table I) is inactive even though it has a $\pi = 1.53$, it is apparent that other requirements for activity exist. The other requirements may be electronic in nature, e.g., the presence of an aromatic ring. However, no reliable correlation with the electronic parameters was found.

With the exception of the cabbage looper, tobacco budworm, and pea aphid (Table II), the phenyl-substituted ester activity was about half the activity of the phenoxy ester. This generally held true when esters of [1,1′-biphenyl]-3-methanol were prepared from other pyrethroid acids (Table III).

As Table III indicates, activity was extremely low or absent in the ([1,1-biphenyl]-3-yl)cyanomethyl esters of the same acids. Other α substituents, methyl-, *n*-butyl-,

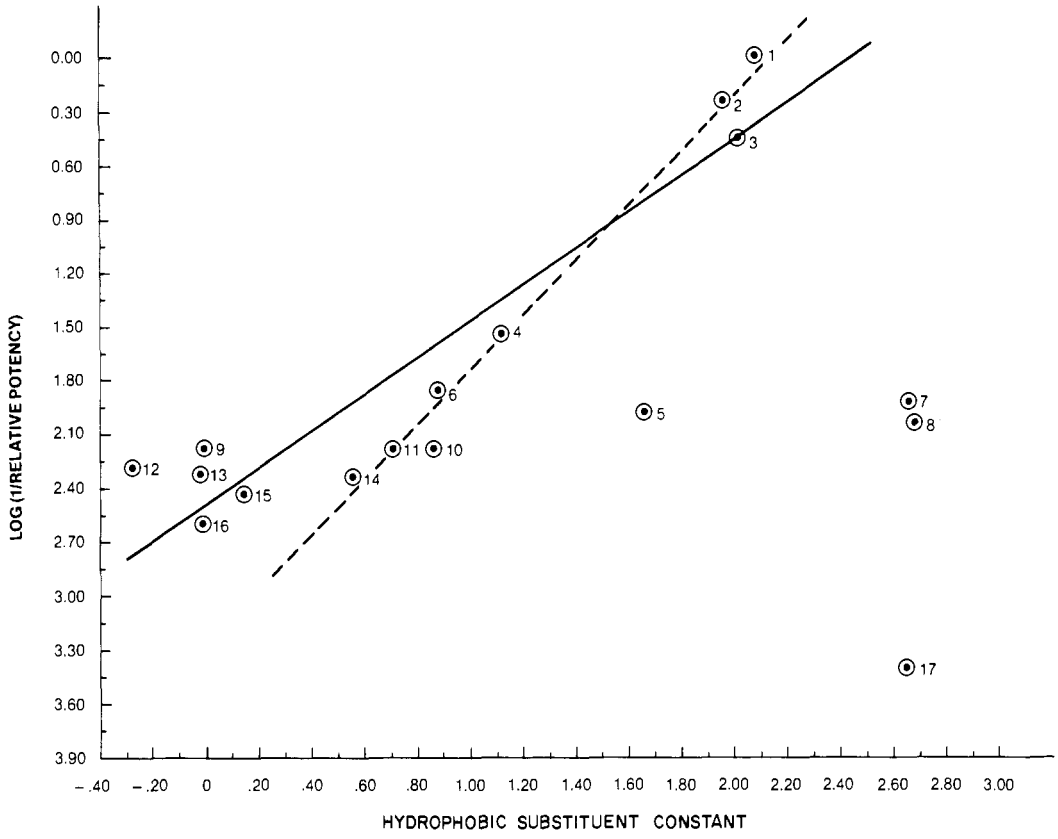
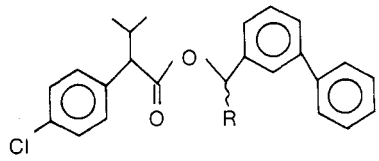
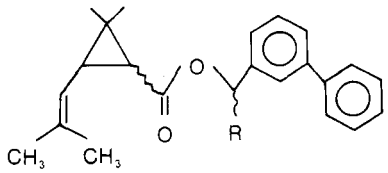
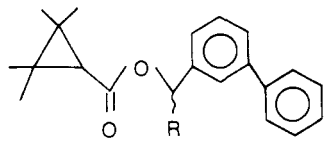
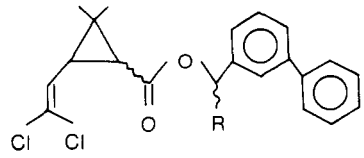


Figure 1. Relationship of meta-monosubstituted hydrophobic substituent constant (π) to Southern armyworm biological response [log [1/(*RP*)]]: (—) eq 5; (- - -) eq 6.

Table III. [1,1'-Biphenyl]-3-Methanol Esters of Common Pyrethroid Acids

	SOUTHERN ARMYWORM TOPICAL RELATIVE POTENCY	
	H	CN
	0.01	Inactive
	0.05	Inactive
	0.02	Inactive
	0.56	0.001

and ethynyl, also resulted in an almost complete loss of activity. It is possible that steric constraints at the active site imposed by the presence of the α substituent lead to poor fit for the biphenyl alcohol.

CONCLUSIONS

The evidence presented indicates that, for meta-monosubstituted benzyl esters, good insecticidal activity can be obtained when the substituent has two centers of unsaturation even if it lacks an atom bridging these centers. The variance in southern armyworm response to such esters is at least partially reflected by differences in the lipophilicity of the meta substituent. This may indicate that the meta substituent plays a part in binding to the active site, perhaps being involved in charge transfer complexation with membrane ion channel proteins. If this is true, then not only does [1,1'-biphenyl]-3-methanol represent a new pyrethroid alcohol but also its esters with pyrethroid acids may provide a new probe for determining the configuration of the active site of pyrethroid insecticides.

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Adsorption–Desorption, Degradation, and Distribution of Permethrin in Aqueous Systems

Mohamed S. Sharom* and Keith R. Solomon

Laboratory studies showed that more than 95% of applied permethrin was adsorbed on lake sediment. Less than 10% of the adsorbed insecticide was desorbed by four 10-mL water rinses. Degradation of permethrin was more rapid in lake water than in flooded sediment, indicating that adsorbed permethrin was more stable than permethrin in the aqueous phase. The cis isomer was more stable toward chemical and biological degradation than the trans isomer. The only major degradation product was *trans*- and *cis*-(dichlorovinyl)dimethylcyclopropanecarboxylic acid. Permethrin applied in aqueous solution on the surface of a sediment column did not penetrate through more than 2 cm of the sediment.

Permethrin [3-phenoxybenzyl (±)-*cis,trans*-3-(2,3-dichlorovinyl)-2,2-dimethylcyclopropanecarboxylate] and other synthetic pyrethroid insecticides have been shown to be more effective against insects and less hazardous to mammals than natural pyrethrins (Abernathy and Casida, 1973). However, they are also more stable and more toxic to fish than the natural pyrethrins (Mauck and Olson, 1976; Zitko et al., 1977). Permethrin is currently being tested against many agricultural and forest insect pests.

Information on the behavior and fate of this insecticide in soil and aquatic systems is essential in assessing its impact on the environment. Recent studies have indicated that permethrin degradation in soil is rapid, with a half-life of approximately 4 weeks or less (Kaufman et al., 1977; Kaneko et. al., 1978; Williams and Brown, 1979). This study reports on the adsorption–desorption of permethrin to a lake sediment, its degradation in water and flooded sediment, and its depth of penetration through sediment columns.

MATERIALS AND METHODS

[*carbonyl*-^{14}C]Permethrin (40:60 cis/trans) with a specific activity of 50 mCi mmol^{-1} was supplied by Chipman, Inc., Canada. The sediment was collected from the top 15 cm at a depth of 5 m of Lake St. George, King City, Ontario, Canada. The sediment contained 43% organic matter as measured by weight loss with ignition at 450 °C and 34% mineral matter after removal of organic materials and carbonates. Clay, silt, and sand represented 48, 34, and 18% of the mineral fraction, respectively. The scintillation cocktail consisted of 0.2 g of POPOP, 10 g of PPO, 666 mL of Triton X-100, and 1334 mL of toluene. Radioassays were carried out in a Nuclear Chicago Unilux II scintillation counter, and counts were converted to disintegrations per min (dpm) by the channels-ratio method. The study was conducted at a room temperature of 21 ± 1 °C.

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Adsorption–Desorption of Permethrin. Two-hundred milligrams (186.56 mg oven-dry weight) of freeze-dried sediment samples was weighed and placed in 15-mL glass tubes. Solutions of permethrin with concentrations of 6.14, 12.52, 24.50, and 41.68 μg L^{-1} were prepared by adding the appropriate amount of radiolabeled compound in 20 μL of acetone to distilled water. For each concentration, triplicate 5-mL aliquots were added to the sediments, and the tubes were covered with aluminum foil lined screw caps. Controls containing only the insecticide solution were included. The tubes were agitated on a wrist-action shaker for 4 h. Preliminary investigation indicated that equilibrium was established within 1 h. The tubes were centrifuged at 3000 rpm for 20 min. After centrifugation, 3.5 mL of the supernatant liquid was pipetted off and permethrin concentration determined. The difference in permethrin concentration between sample and control tubes was attributed to adsorption on sediment.

The same sample tubes (excluding the tubes that contained the highest permethrin concentration) were used for determining successive desorption of permethrin from sediment. After removal of 3.5 mL of the supernatant, 8.5 mL of distilled water was added to each tube to make a total aqueous volume of 10 mL. The tubes were capped and agitated for 4 h to establish a new equilibrium. The tubes were then centrifuged and 8.5 mL of the supernatant was pipetted off. Two milliliters of the pipetted supernatant was used to determine permethrin concentration. Another 8.5 mL of distilled water was then added to each tube, and the desorption process was repeated another 3 times. The amount of insecticide remaining adsorbed to the sediment after each desorption process was calculated and expressed as percent of the initial amount adsorbed to the sediment.

Degradation of Permethrin in Water and Flooded Sediment. Five-hundred milliliters of radiolabeled permethrin solution (15 μg L^{-1}) was made in lake water (pH 7.8). Half of this solution was treated with sodium azide (0.2% w/w). Five-milliliter aliquots of the untreated and

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