Volatilization of Insecticides from Various Substrates

E. Paul Lichtenstein* and Kenneth R. Schulz

The volatilization of four chlorinated hydrocarbon and four organophosphorus insecticides from various substrates over a 24-hr period was investigated. In addition, the volatility of Dyfonate, applied at increasing concentrations to water, was studied over a 24-hr period, and at a fixed concentration over a 10-day period. The rate of volatilization of the various insecticides was a function of the properties of a particular chemical and of the substrate to which the insecticide had been applied. The most volatile insecticide was aldrin, while azinphosmethyl was least. Highest volatilization rates were observed from water while relatively small amounts of insecticides volatilized from soils. Addition of soil, algae, or a detergent to water reduced the volatility of a specific compound in many cases. With increasing concentrations of Dyfonate in water, increasing amounts of the insecticide volatilized from water that had been treated with the insecticide at 2.5 to 20 ppm. However, at higher concentrations (40 to 160 ppm) no increase in volatilization occurred. During a 10-day incubation period, increasing amounts of Dyfonate and two unidentified metabolites volatilized from water.

Since pesticidal chemicals have been used in agriculture for pest control, intensive research has been conducted to obtain information relative to the ultimate fate of these chemicals once they have been applied to a specific target area. During the initial 10-yr period after the introduction of these chemicals, analytical methods were not as sensitive as they are today. Quite often nondetectability of a particular compound has been equated with "disappearance" or "loss" of the originally applied pesticide. As research in this field intensified, however, it became evident that pesticidal residues "disappear" through metabolism into other compounds or are transported from the originally treated or contaminated target area such as soils and others. One kind of such movement occurs through volatilization of the chemical. Bowman *et al.* (1959) reported the loss of DDT from aqueous solutions, and Lichtenstein and Schulz (1961) showed that aldrin was lost from soils through volatilization at a rate which was directly affected by soil moisture. Water had apparently caused a displacement of aldrin from soil particles, thus facilitating volatilization of the insecticide. Harris and Lichtenstein (1961) described various factors that affect volatilization of insecticide residues from soils under laboratory conditions, while it was shown in field experiments (Lichtenstein *et al.*, 1962) that cover crops, such as alfalfa, considerably reduced the loss of aldrin and heptachlor from a silt loam soil. It was in 1963 (Lichtenstein *et al.*) that the disappearance of aldrin from sterile nutrient agar was reported while the presence of microorganisms retarded this disappearance. Later (Lichtenstein *et al.*, 1968) it was demonstrated that this loss of aldrin or dieldrin occurred through volatilization because the vapors could be trapped on oil impregnated filter paper. Fungi grown on insecticide treated media reduced this volatilization. Wheeler (1969) investigated the volatilization of dieldrin from aqueous algae cultures.

In this study the volatilization of four chlorinated hydrocarbon and four organophosphorus insecticides as affected by various substrates was investigated under controlled laboratory conditions. The quantitative relationships between insecticide concentrations and volatilization, as well as volatilization of an insecticide with time, were also studied.

Department of Entomology, University of Wisconsin, Madison, Wis. 53706

* To whom correspondence should be addressed.

MATERIALS AND METHODS

Chemicals. Analytical grade insecticides were used in both nonradioactive and C^{14}-labeled forms. They are listed in Table I together with their vapor pressures and water solubilities. Unfortunately, vapor pressure data for all insecticides at one temperature were not available. Carbon^{14}-labeled aldrin and dieldrin were obtained from the Shell Chemical Co., *p,p'*-DDT and lindane from Amersham/Searle, ethyl parathion from Tracerlab, Dyfonate (*O*-ethyl-*S*-phenyl-ethyl phosphonodithioate) from the Stauffer Chemical Co., azinphosmethyl (Guthion) from the Chemagro Corp., and diazinon from the Geigy Chemical Corp. LAS (linear alkyl benzene sulfonate), a biodegradable detergent, was obtained from the Atlantic Refining Co. The formulation consisted of 92% LAS, 7% sodium sulfate, and 0.5 to 1% moisture. Ethanol (95%), diethyl ether, and redistilled hexane were used as solvents.

Insecticide Substrates. To test the effects of various substrates on the rate of volatilization of insecticides, glass beads (150-μ diameter), water, or a silt loam soil were treated with the different toxicants. In addition, the effects of soil, algae, or detergents in water or of detergents in soil were investigated. To obtain finely suspended soil particles in water, 250 g of a loam soil were mixed for 5 min with 1 liter of tap water in a Lourdes homogenizer. After standing for 18 hr, the water was decanted and is referred to in this paper as "soil-water." Algae (*Euglena gracilis*) were grown in nutrient media ("Euglena Broth," Difco Laboratories, Detroit) and were used at a density of 6.4×10^6 cells per ml. A detergent which had been found to increase the presistence of some insecticides in soils (Lichtenstein, 1966) was tested for its effects on the rate of insecticide volatilization. For this purpose, LAS was added at 0.1% to a buffer solution (sodium phosphate buffer, 0.1*M*, pH 7.0) or to soil. Application of LAS was performed as previously described (Lichtenstein, 1966).

A total of eight experimental substrates was finally obtained: glass beads, tap water, soil water, algae in nutrient media, buffer solution, buffer solution containing 0.1% LAS, silt loam, and silt loam containing 0.1% LAS.

Application of Insecticides. Three grams of glass beads, 2 ml of one of the liquid substrates, or 2 g of soil were placed into 50 ml Erlenmeyer flasks which had a ground neck joint (19/22 standard taper). Each toxicant was then applied by pipetting 25 μl of ethanol containing 25 μg (0.05 μc) of C^{14}-

Table I. Physical Properties of Insecticides

Insecticide	Vapor pressure mm Hg (°C)	Water solubility ppm
Aldrin[a]	4.9×10^{-5} (30° C)	0.01
Dieldrin[a]	1.4×10^{-6} (30° C)	0.10
p,p'-DDT[b]	3×10^{-7} (25° C)	0.001
Lindane[c]	9.4×10^{-6} (20° C)	6.6
Dyfonate[d]	2×10^{-4} (25° C)	13
Parathion[e]	2.3×10^{-5} (30° C)	24
Azinphosmethyl[e]	$< \times 10^{-7}$	34
Diazinon[e]	2.8×10^{-4} (30° C)	40

[a] Porter (1965). [b] Bowman *et al.* (1960). [c] Robeck *et al.* (1965). [d] Menn (1969). [e] Schrader (1963).

labeled insecticide onto each substrate within the flask. To facilitate further the distribution of each of the insecticides in or on the solid substrates, 1 ml of diethylether was also added to glass beads or soils. After the ether had evaporated, insecticide vapor traps were placed onto each of the Erlenmeyer flasks.

Insecticide Vapor Traps and Incubation. Vapor traps were prepared by treating 0.5 g of borosilicate glass wool with 1 ml of 5% corn oil in hexane. After evaporation of the solvent at 50° C, one glass wool plug was placed into a 5 cm long glass tube (1.6 cm diameter) that had a standard taper joint of 19/22. (In preliminary experiments two plugs on top of each other were also tested.) After that the trap was placed onto the Erlenmeyer flask containing the treated substrate, the joint was sealed with phosphoric acid, and the tube was covered with aluminum foil.

Flasks and traps were then placed into a metabolic water bath shaker (New Brunswick Scientific Co.) and incubated at 30° C at the slow shaking rate of 50 revolutions per min. Incubation times were 24 hr in Experiments 1 and 3, and up to 10 days in Experiment 4.

Extraction and Analyses. At the end of the incubation period, each trap was removed from the substrate containing flasks and placed onto another flask. Two portions of 10 ml of hexane were then poured through the glass wool. To expel all the remaining hexane, air was finally passed through the plug. This resulted in a total recovery of 19.4 to 19.8 ml of hexane in the collection flask.

Analyses of these hexane extracts was performed by liquid scintillation counting (LSC) or by gas-liquid chromatography (glc). For LSC, 2 ml portions of the hexane extracts were added to 13 ml of scintillation mixture (6 g of PPO, 0.25 g of dimethyl POPOP in 1000 ml of toluene) and counted for 10 min in a Model 3320 Packard Tri-Carb scintillation spectrometer. Data were corrected for background, counter efficiency, and dilution, and finally expressed as radioactivity in percent of the applied dosage.

For analyses by glc, extracts from three replicated samples were pooled and analyzed with a Packard Model 7834 gas chromatograph. The instrument was equipped with a 150 mCi tritium electron affinity detector operated at 50 V. The glass column (1.22 meters, 3 mm i.d.) contained a preconditioned 1 to 1 mixture of 5% QF-1 and 5% DC-200 coated on 80 to 90 mesh Anachrom AS. A column pressure of 17 psi of nitrogen resulted in a flow rate of 120 ml per min. The injector temperature was held at 260° C, the column oven at 170° C, and the detector at 215° C.

After glass wool plugs had been extracted with hexane, they no longer contained measurable amounts of radioactivity, nor could any radioactivity be detected on the inner surfaces of the aluminum foil which covered the glass tubes during the 24-hr incubation time. Preliminary experiments also indicated that a second upper glass wool plug did not contain radioactivity. It appeared from these data that the oil impregnated traps served as efficient vapor barriers.

The efficiency of the vapor traps and of the extraction procedure was tested in preliminary experiments. For this purpose 2 ml of soil water were placed into each of 24 50-ml Erlenmeyer flasks. Each of the eight insecticides was then added (25 μg per 2 ml water) to the soil water in three flasks, thus resulting in eight tests. Traps were then placed onto these flasks, followed by incubation of this closed system at 30° C for 24 hr. The insecticide was then extracted from the vapor traps as described. The soil water in the flasks was quantitatively transferred with 12 ml of counting solution (4 g of PPO, 0.05 g of dimethyl POPOP, 120 g of naphthalene, and 880 ml of 1,4-dioxane) into counting vials for radioassay. The total radioactivity (average of values obtained with eight insecticides) recovered in this way from the insecticide-treated soil/water and vapor traps was 93.2 ± 1.6% of the applied dosage.

Experiment 1. Volatilization of Insecticides from Various Substrates. To determine the rate of volatilization of the insecticides from the different substrates, 3 g of glass beads, 2 ml of each of the liquid substrates, or 2 g of a moist silt loam soil were treated with each of the insecticides described. Experiments with one insecticide were replicated three times, thus resulting in 24 flasks containing eight different substrates. Flask and traps were then incubated for 24 hr as described, followed by extraction of the trapped vapors and analyses by both LSC and glc.

Experiment 2. Vapor Toxicity of Insecticides. To test the toxic effects of insecticidal vapors, fruit flies (*Drosophila melanogaster* Meig) were placed above insecticide treated soil water. For this purpose 5 ml of soil water was pipetted into each of 18 4-oz bioassay jars (5.5 cm i.d., 6 cm deep) (Edwards *et al.*, 1957) and 50 μl of ethanol containing 50 μg of one of the eight insecticides were added to the water in two jars, resulting finally in eight duplicated tests and one control. Copper screen cages then were inserted into the jars in such a way that the bottom of each cage was 2 to 3 mm above the water surface. Fifty 3-day old *Drosophila* flies were then placed within the cage of each jar and held for 24 hr at room temperature. In this way the flies did not come into contact with the insecticide-treated substrate and any observed mortalities would have been due to insecticide vapors. Controls were conducted with soil-water that had been treated with ethanol only. Mortality counts were performed periodically over a 24-hr period.

Experiment 3. Effect of Dyfonate Concentration on Volatility. The rate of loss of a particular insecticide from a given substrate depends on a variety of factors. One of these is the concentration of the insecticide itself, as was shown by Lichtenstein and Schulz (1959) with soils. They reported that under both field and laboratory conditions, the relative loss of an insecticide from soils, expressed in percent of the applied dosage, was to some extent inversely proportional to the insecticide concentration.

To test this observation, experiments were conducted with increasing concentrations of Dyfonate in soil-water, and volatilization of the insecticide was measured. To obtain a required concentration of the insecticide in 2 ml of soil-water, a specific amount of C^{14}-Dyfonate in hexane was pipetted onto the bottom of each of three 50-ml Erlenmeyer flasks. After careful evaporation of the hexane, 50 μl of

Table II. Volatilization of Insecticides from Various Substrates at 30° C

Substrate		Volatilized in % of applied insecticide/24 hr							
		Aldrin	Dieldrin	*p,p'*-DDT	Lindane	Dyfonate	Parathion	Azinphosm.	Diazinon
Glass beads	LSC[a]	20.5 ± 1.0	0.48 ± 0.15	0.15 ± 0.01	13.5 ± 0.40	10.3 ± 0.30	0.35 ± 0.10	<0.01	1.63 ± 0.26
	GLC[b]	19.4	0.44	TR[c]	9.80	7.80	TR	ND[d]	TR
Tap water +									
None	LSC	24.6 ± 0.4	1.31 ± 0.05	0.90 ± 0.08	16.4 ± 0.06	15.2 ± 0.20	0.96 ± 0.09	ND	1.93 ± 0.38
	GLC	23.3	1.1	0.88	10.4	15.0	0.90	ND	TR
Soil[e]	LSC	26.1 ± 0.4	1.32 ± 0.05	0.78 ± 0.09	11.5 ± 0.60	16.3 ± 1.1	0.96 ± 0.25	<0.01	0.63 ± 0.28
	GLC	23.8	1.1	0.80	11.0	14.0	0.80	ND	ND
Algae[f]	LSC	25.1 ± 0.9	0.95 ± 0.01	0.76 ± 0.18	5.5 ± 0.22	5.4 ± 0.30	0.68 ± 0.11	<0.01	0.52 ± 0.28
	GLC	21.5	0.88	0.88	4.10	4.0	0.60	ND	ND
Buffer soln +[g]									
None	LSC	25.4 ± 0.9	0.85 ± 0.22	0.95 ± 0.06	15.7 ± 1.10	20.5 ± 1.10	1.23 ± 0.15	0.02 ± 0.01	0.99 ± 0.28
	GLC	21.9	0.68	0.74	12.5	17.0	1.60	...	TR
LAS 0.1%	LSC	10.6 ± 0.3	1.70 ± 0.07	0.82 ± 0.06	9.50 ± 0.30	11.7 ± 0.92	1.67 ± 0.28	0.02 ± 0.01	0.64 ± 0.08
	GLC	9.10	1.70	0.72	5.40	11.0	1.50	...	TR
Soil +									
None	LSC	3.3 ± 0.2	0.28 ± 0.04	0.08 ± 0.05	0.92 ± 0.03	1.0 ± 0.50	0.07 ± 0.02	ND	0.20 ± 0.08
	GLC	2.8	0.27	TR	0.65	0.80	TR	ND	ND
LAS 0.1%	LSC	2.4 ± 0.1	0.15 ± 0.02	0.20 ± 0.13	0.98 ± 0.17	1.2 ± 0.10	0.07 ± 0.02	ND	0.10 ± 0.01
	GLC	1.80	0.16	0.29	0.73	0.80	TR	ND	ND

[a] LSc: C-14, determined by liquid scintillation counting, average of three replicates. [b] Glc: determined qualitatively and quantitatively by gas-liquid chromatography. [c] TR = Trace. [d] ND = not detectable. [e] Loam soil particles suspended in tap water. [f] Algae cells (*Euglena gracilis*) suspended in nutrient media. [g] 0.1 *M* sodium phosphate buffer, pH 7.0.

ethanol were added to redissolve the insecticide before 2 ml of soil-water were also added. In this way concentrations of 2.5, 5, 10, 20, 40, 80, or 160 ppm of Dyfonate in soil water were finally obtained.

Previously prepared vapor traps were placed onto the flasks, which were then incubated at 30° C for 24 hr as described. Afterwards the glass wool plugs were extracted and analyzed by LSC. This particular experiment was conducted twice, each time with three replicates of a given insecticide concentration in soil water. Results were finally averaged and expressed in μg of Dyfonate that had volatilized in relation to the applied dosage.

Experiment 4. Volatilization of Dyfonate With Time. To test the rate of loss of Dyfonate from soil-water over a specific time period, 2 ml of soil-water were pipetted into each of 24 50-ml Erlenmeyer flasks. After treatment of each water sample at 12.5 ppm with C^{14}-Dyfonate, vapor traps were placed onto each flask, and incubation at 30° C was started. One, 2, 3, 4, 5, 6, 8, and 10 days later three flasks were removed from the shaker. The respective glass plugs were then extracted and analyzed by both LSC and glc. After these data has been obtained, the extracts were also analyzed by thin-layer chromatography (tlc) and autoradiography. For this purpose the pooled replicate hexane extracts were concentrated in a flash evaporator at 25° C. Each concentrate was then subjected to the sweep cleanup procedure (Storherr and Watts, 1965) in order to separate the insecticide or its potential metabolites from the oil. The residues were passed through glass wool packed sweep columns and eluted with redistilled benzene-acetone (3 to 1) at 190° C.

These cleaned up concentrates—obtained at different incubation time intervals—were then chromatographed on silica gel-coated thin-layer plates, using chloroform-ethyl acetate (1 to 1) as the solvent system (Menn, 1969). Developed plates were exposed for 60 hr to X-ray film sheets (Ansco High Speed). Based on the spots that became visible after development of the X-ray film, four silica gel areas (R_f 0.79 to 0.64, 0.64 to 0.43, 0.43 to 0.34, and 0.34 to 0.00) were scraped off the plate and tested for radioactivity. For this purpose the isolated silica gel areas were scraped into counting vials to which 12 ml of scintillation liquid (4 g of PPO, 0.05 g of POPOP, 120 g of naphthalene, and 880 ml of 1,4-dioxane) containing 4% Cab-O-Sil (Godfrey L. Cabot, Inc., Thixotropic Gel Powder) were added. After thorough mixing, liquid scintillation counting was performed as described.

RESULTS AND DISCUSSION

Experiment 1. Volatilization of Insecticides from Various Substrates. Insecticide volatilization from eight different substrates was measured after a 24-hr incubation period. The amounts volatilized during that period were expressed in percent of the applied dosages and are presented in Table II. It is evident from these data that the rate of volatilization of the various insecticides was a function of the properties of a particular chemical and of the substrate to which the insecticide had been applied. Since results obtained by glc were generally in close agreement with those obtained by LSC, it appears that the compounds which had volatilized were primarily in the form of the originally applied chemical. In general, one might assume that low water solubility and a relatively high vapor pressure would enhance volatilization from aqueous substrates. But no direct relationships could be established between the water solubility of a particular compound and its volatilization, although the chlorinated hydrocarbon insecticides have a water solubility (Table I) which is below the applied dosage of 12.5 ppm and the organophosphorus compounds have a water solubility which is above that figure. The most volatile insecticide tested was aldrin, followed by lindane, Dyfonate, dieldrin, parathion, DDT, diazinon, and azinphosmethyl. The latter compound hardly volatilized, since only traces of radioactivity could be found in some of the vapor traps. This could have been the result of its relatively high water solubility of 34 ppm and its low vapor pressure.

Highest volatilization rates were observed from water, while relatively small amounts of insecticides volatilized from soils. For example, 7.7 times more aldrin volatilized from water than from soil. This figure was 4.6 with dieldrin, 11 with DDT, 17.5 with lindane, 15 with Dyfonate, 14 with parathion, and 9.6 with diazinon. Addition of soil, algae, or detergents to water reduced the volatility of a specific com-

Table III. Vapor Toxicity of Insecticides Volatilized from Soil Water[a] at Room Temperature

Insecticide	Insect Mortalities[b] LT-90[d]	Mort/3h[e]	μg/24h[c]
Aldrin	2.5	98	5.95
Dieldrin	15.0	0	0.28
p,p'-DDT	>24[f]	0	0.20
Lindane	2.0	97	2.75
Dyfonate	1.0	92	3.50
Parathion	20.0	5	0.20
Azinphosmethyl	>24	0	ND
Diazinon	8	5	ND
None	>24	0	

[a] Standing tap water with suspended loam soil particles. [b] *Drosophila melanogaster* exposed to insecticide vapors. [c] Micrograms of insecticide volatilized at 30° C from slowly agitated soil water as determined by gas-liquid chromatography (Table II). [d] Hours, in which 90% mortality of insects was obtained. [e] Percent mortality of insects after a 3-hr exposure period. [f] No mortality of insects during a 24-hr exposure period. ND = nondetectable.

pound in many cases. Thus, soil particles in water reduced particularly the volatilization of DDT and lindane, while algae in water reduced the volatilization of lindane and Dyfonate by 60%. LAS in soil reduced the volatilization of aldrin and dieldrin.

Experiment 2. Vapor Toxicity of Insecticides. After *Drosophila* flies had been exposed to vapors that emanated from insecticide treated soil water, quantitative differences in toxic effects were observed (Table III). The approximate time that was required to cause a 90% mortality of the flies (LT-90) varied considerably for each insecticide The most toxic effects were observed with Dyfonate in soil water (LT-90 = 1 hr), followed by lindane, aldrin, dieldrin, diazinon, parathion, DDT, and azinphosmethyl. However, when the actual amounts of insecticides that had volatilized during a 24-hr incubation period (Table III—μg/24h) were compared with the observed toxicity data, no direct correlation could be established. This was not surprising, since the mortalities of insects are not only dependent on the actual amounts of insecticide vapor that come into contact with the insect, but also on the specific toxic action of a particular insecticide. All that was shown in this experiment is the volatilization of toxic vapors from insecticide treated soil-water. DDT, which is relatively nontoxic to the strain of *Drosophilia* (Cinnebar) used, did not cause any mortality during the 24-hr exposure period, although measurable amounts of this insecticide had volatilized during that time. Azinphosmethyl did not cause insect mortality either, but this was the result of its extremely low volatilization rate.

Experiment 3. Effect of Dyfonate Concentration on Volatility. Since it had been reported that the rate of loss of insecticides from soils was to some extent inversely proportional to their concentration in soils (Lichtenstein and Schulz, 1959), it was interesting to note that similar results were obtained when the rate of volatilization of Dyfonate was compared to its original concentration in the substrate. The amounts of C^{14}-Dyfonate volatilized from soil-water during a 24-hr incubation period are presented on a log-log basis in Figure 1. The insecticide was lost through volatilization in increasing amounts from water that had been treated with Dyfonate at 2.5, 5.0, 10, and 20 ppm, resulting in a linear relationship as shown in Figure 1. The relative amounts, however, expressed in percent of the initial insecticide concentration in the water (2.5 to 20 ppm) did not change considerably and ranged from 21 to 16% (Figure 1—"in % of applied").

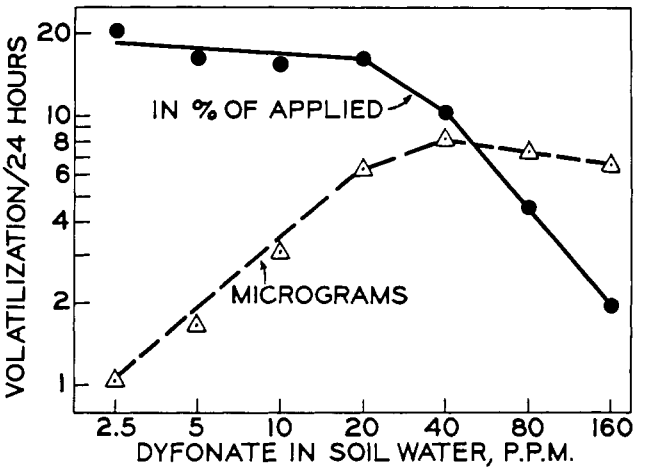

Figure 1. Effect of C^{14}-Dyfonate[R] concentrations in soil-water on the volatilization of C^{14}-containing compounds during a 24-hr incubation period at 30° C

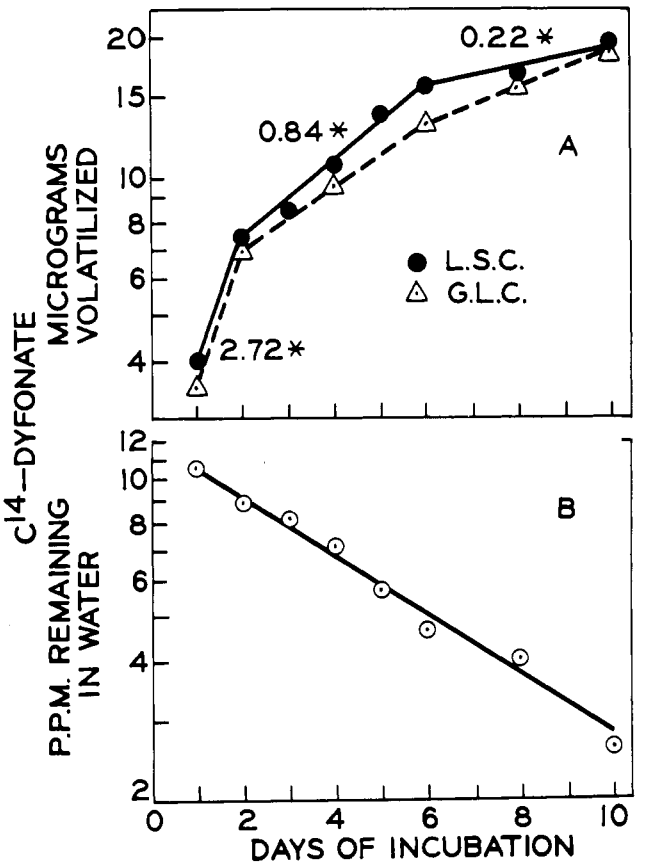

Figure 2. Volatilization of C^{14}-Dyfonate from soil-water at different time periods. A = results obtained by liquid scintillation counting (LSC) and gas-liquid chromatography (glc). * = slope. B = results calculated from LSC data in Figure A above

However, as insecticide concentrations in the water increased, the actual amounts volatilized no longer increased. Thus, 8.28 ± 0.68 μg of C^{14}-Dyfonate were recovered from the vapor traps over soil water that had been treated with the insecticide at 40 ppm and only 7.40 ± 0.82 μg or 6.70 ± 0.27 μg from traps over water that had been treated with Dyfonate at 80 and 160 ppm, respectively. When these data were expressed in percent of the applied insecticide dosage it became evident that 16.1 ± 1.7% of the applied dosage, (40 μg/2 ml soil water) had volatilized from a Dyfonate con-

Table IV. Volatilization of C^{14} from Dyfonate Treated Soil Water and Isolation of C^{14} by Thin-Layer Chromatography

Area, R_f[a]	CPM recovered from vapor traps, after days of incubation at 30° C					Dyfonate standard[b]
	2	4	6	8	10	
0.79–0.64	46,405	10,342	67,792	84,519	82,301	87,837
0.64–0.43	91	174	196	246	212	135
0.43–0.34	257	284	502	951	643	204
0.34–0.00	1,130	1,639	2,101	3,501	2,609	838

[a] Silica gel areas isolated from thin-layer plates for the determination of radioactivity. Clearly visible spots at R_f 0.75, 0.39, and 0.00 were obtained by X-ray autoradiography during $2\frac{1}{2}$ days of exposure. [b] CPM obtained after passing of 25 μg of C^{14}-Dyfonate with oil through sweep cleanup procedures and tlc, Dyfonate, R_f = 0.75.

centration of 20 ppm and 10.3 ± 0.8%, 4.6 ± 0.5%, or 2.0 ± 0.0% from concentrations of 40, 80, and 160 ppm, respectively. This then indicated that the relative amount of Dyfonate that volatilized from soil water was to some extent inversely related to its concentration in the water. This might also demonstrate that the "half life" of a particular insecticide is not absolute and is, among other factors, also dependent on the insecticide concentration in a given substrate.

Experiment 4. VOLATILIZATION OF DYFONATE WITH TIME. The amounts of C^{14}-containing compounds volatilized from C^{14}-Dyfonate-treated soil water after 1, 2, 3, 4, 5, 6, 8, or 10 days of incubation increased with time. The rate of volatilization, though, declined with time, as indicated by the different slopes in Figure 2A. This rate was greatest during the first 2 days of incubation (slope = 2.72) and smallest during the 6 to 10 day incubation period (slope = 0.22). Fifty percent of the applied radioactivity had volatilized after 4.8 days. After 10 days, the concentration of Dyfonate in water amounted to only 2.56 ppm, and had, therefore, dropped to 20% of the applied dosage.

Data obtained by glc were specific for Dyfonate and showed that in all cases its amount was slightly lower than that which had been determined by LSC. This indicated that C^{14}-containing compounds other than Dyfonate could have volatilized and accounted for the differences in recoveries as determined by LSC and glc. Using tlc and autoradiography procedures as described, the presence of these compounds was indeed demonstrated (Table IV). Twenty-five μg of Dyfonate standard, passed with oil through the described sweep cleanup procedure, yielded only one visible spot (R_f 0.75) by autoradiography, although relatively small amounts of radioactivity were also detected by LSC of the other three silica gel areas. However, analyses of extracts of vapor traps revealed the presence of other compounds in addition to Dyfonate. Although 96 to 98% of the recovered radioactivity had the same R_f value as Dyfonate (R_f 0.75), two clearly visible additional spots (R_f 0.39 and 0.00) appeared on the X-ray film during its 60-hr exposure to the thin-layer plates. Since the radioactivity of these spots increased up to 8 days and was also considerably higher than in the 25 μg Dyfonate standard, it appeared that more polar metabolites had been formed from Dyfonate.

The total amount of radioactivity recovered from the thin-layer plates increased daily, although no attempt was made to evaluate these data quantitatively. It is quite possible that during the process of extract concentration, sweep cleanup, and tlc procedures, radioactivity was lost, as indicated by the data that were obtained with extracts after 4 days of incubation.

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