

ADSORPTION, DESORPTION, SOIL MOBILITY, AQUEOUS PERSISTENCE AND OCTANOL-WATER PARTITIONING COEFFICIENTS OF TERBUFOS, TERBUFOS SULFOXIDE AND TERBUFOS SULFONE

KEY WORDS: insecticide, solubility, leaching, stability, degradation

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ABSTRACT

Terbufos, t. sulfoxide and t. sulfone ($5 \mu\text{g ml}^{-1}$) were incubated in natural, sterilized natural and distilled water, with initial pH values of 8.8, 8.8 and 6.0, respectively, at 20°C . First-order disappearance was observed for the three compounds. Rates in natural and sterilized water were similar indicating chemical degradation predominated. Terbufos disappeared rapidly ($t_{1/2} = 3$ days) in all systems. T. sulfoxide and t. sulfone were more persistent in the natural ($t_{1/2} = 18-40$ days) and distilled water ($t_{1/2} = 280-350$ days). Adsorption data for the three compounds in four soil-water systems showed the decreasing order of adsorption to be terbufos » t.sulfoxide \approx t.sulfone. Desorption from soils fortified at 5 g g^{-1} with water was examined for 4 successive 18-hr cycles. T.sulfoxide and t. sulfone were totally desorbed; terbufos was too unstable to study. The mobility of the compound in soil eluted with water was in the order, t.sulfoxide \approx t.sulfone » terbufos, in agreement with adsorption-desorption results. The octanol-water partitioning coefficients for terbufos, t. sulfoxide and t. sulfone, at 23°C , were 3.30×10^4 , 164, and 302, respectively.

INTRODUCTION

The behavior of pesticides in the environment is governed by numerous physicochemical processes, which for soil-water systems include partitioning (adsorption, desorption), movement (mass flow, diffusion) and degradation (hydrolysis, redox reactions). It is of importance, from both a cost effectiveness and environmental safety point of view, that the ultimate disposition of a pesticide (to the first non-toxic breakdown product) in a soil-water system be well understood.

Terbufos, an organophosphorus insecticide, is recommended for control of corn rootworm in Ontario. It has been shown to be relatively non-persistent in soil, being converted to t. sulfoxide and subsequently to t. sulfone^{7,14}. Terbufos adsorption in soil exhibits a strong positive correlation with organic matter¹⁰ and consequently is not as mobile in soil^{11,12} as the more soluble sulfoxide and sulfone⁵. The desorption behavior of terbufos has been reported¹⁰, but the conclusions presented are suspect because "single-cycle" desorption isotherms were generated rather than the correct desorption isotherms derived by successively desorbing from a single initial concentration^{2,23}.

The purpose of this study was to investigate the adsorption, desorption and mobility behavior of terbufos, t. sulfoxide, and t. sulfone using common Ontario agricultural soils, and to examine the persistence of these compounds in water using previously developed experimental procedures¹⁶. Octanol-water partitioning coefficients for the three compounds were also determined.

MATERIALS AND METHODS

Adsorbents. Three soils and a stream sediment (the latter included as a mineral soil), the properties of which are shown in Table 1, were used as adsorbents. The soils were air-dried, crushed to pass a 2 mm sieve, and thoroughly mixed before use.

Compounds. Terbufos, t.sulfoxide, and t.sulfone were supplied by American Cyanamid, Princeton, N.J., and had purities of 97.2, 95+ and 95+%, respectively. Reagent grade octanol used in the partitioning studies was distilled over sodium hydroxide.

TABLE 1

Characteristics of the Soils and Stream Sediment.

	Percent				pH*
	sand	silt	clay	organic matter	
Plainfield Sand	91.5	1.5	7	0.7	6.9
Big Creek Sediment	71	22	7	2.3	6.5
Bondhead Sandy Loam	77	15	8	3.9	6.9
Muck Soil	52	34	14	36.7	6.3

* measured in 0.01 M CaCl₂ (20 ml to 10 g soil)

Adsorption Studies. Batch-type adsorption experiments, details of which have been described, were used to generate adsorption data. Triplicate one-gram soil samples in 30-ml aliquots of aqueous insecticide solution (five concentrations) were tumbled for 18 hr at 20±1.5°C for equilibration prior to centrifuging at 34800 x g for 0.5 hr and subsequent sampling.

Desorption Studies. The four soil adsorbents were spiked at 5.0 µg⁻¹ g (oven-dry basis) in a hexane-acetone (95:5 v/v) solvent using the procedure described elsewhere¹⁶. Triplicate samples of treated soil were weighed into Corex centrifuge bottles and 30-ml aliquots of distilled water were added. After shaking, followed by centrifuging, 25 ml was removed for analysis and was replaced with a fresh 25 ml aliquot of water for the next cycle. Four such cycles were completed. Samples of treated soils were extracted with CHCl₃, the CHCl₃ removed and replaced with 95% hexane-acetone solvent. The samples were then analyzed to confirm treatment rates and to account for any degradation. Terbufos desorption was not determined because of its unstable nature during the several days required for the experiment. Errors in determining the aqueous concentration in the early desorption cycles would be magnified and passed on to successive cycles, leading to unacceptable accuracy.

Octanol-Water Partitioning Coefficients (K_{OW}). Purified octanol and distilled water (re-distilled over $KMnO_4$) reagents were mutually saturated using separatory funnels before use. Ten ml of an appropriate concentration of each insecticide in octanol (in triplicate) was pipeted into 60 ml separatory funnels. Forty ml of water was added, the funnel was shaken for 30 sec. and was allowed to sit overnight. A 25-ml aliquot of the aqueous layer was taken for centrifugation at $34,800 \times g$ for 1 hr at $23^\circ C$. Aliquots were taken for analysis by GLC. Usually, three successive equilibration-extraction cycles were sufficient to produce constant K_{OW} values.

Mobility Studies. The apparatus used to study insecticide mobility in soils has been described²². The treated soil (insecticide applied in hexane solution and the solvent removed) was held in a glass cylinder (41 mm I.D. x 65 mm high) set in fine sand, which in turn was set inside a vacuum filtration funnel. Ten grams of treated soil (at $5 \mu g g^{-1}$ insecticide, oven-dry basis) was overlain by untreated soil (20 g for mineral soils, 10 g for muck) to prevent the treated soil sample from being disturbed by the influx of water. Two liters of distilled water were drawn through the treated soil (cross-sectional area, 13.2 cm^2) by suction in approximately 2.5 hr, and collected as 10 x 200-ml fractions. This gave a linear flow rate of approximately 60 cm hr^{-1} . Samples of treated soil and the soil core left after leaching were extracted (as per the Desorption Section) and analyzed to confirm initial treatment levels and to determine whether any insecticide remained. Each leaching experiment was conducted in duplicate.

Water Persistence Studies.

Natural or Distilled Water. Terbufos or t.sulfoxide or t.sulfone) dissolved in $CHCl_3$, was pipeted into 1-liter screw cap glassbottles, and the solvent was evaporated. Either one liter of distilled water (pH 5.50) or natural water (pH 7.93) from the Holland Marsh, Ontario drainage system¹⁷, was then added to the bottle containing the insecticide sample. After

mixing, 2 x 400 ml aliquots (duplicates for analytical purposes) were taken from the 1-liter bottle and stored in darkness at 20°C. Appropriate aliquots were removed from these duplicates on successive sampling dates. The nominal treatment level in all water persistence studies was 5 g/ml.

Sterilized, Natural Water. Natural water was sterilized by autoclaving in Pyrex beakers. Two ml of chloroform containing 250 g of insecticide was pipeted into sterile 112-ml screw cap glass bottles and the solvent was evaporated. Fifty ml of sterilized water was added and the samples were stored at 20°C in darkness. All operations with sterile samples were performed in an environmental air control cabinet.

At 0 day and at 1,2,4,9, and 16 wk intervals, aliquots were removed from all treatments for analysis. To avoid contamination, samples in sterilized natural water were taken from previously unopened bottles at each sampling date.

Analytical. Automated gas-liquid chromatographs (GLC) equipped with alkali-flame ionization detectors were used for analysis as reported previously⁵. All samples in the adsorption, desorption, and octanol-water partitioning studies were injected in aqueous methanol solvent, and standards were prepared in the same solvent as previously reported¹⁶. All three compounds were quite stable in the aqueous methanol solvent. In the mobility and persistence studies, samples were extracted with chloroform, the chloroform evaporated, and the sample made to volume in 10% acetone in hexane. This technique provided lower detection limits, where required, than the above-mentioned aqueous methanol technique, but was much more time consuming. The glass GLC columns, 0.46 x 2 mm I.D., were packed with 80/100 mesh Chromosorb W, AW DMCS, coated with 1% OV-275 and treated with 0.1% H₃PO₄. Operating column temperatures were 100°C for terbufos and 155-170°C for t.sulfoxide and t.sulfone.

RESULTS

Water Persistence Studies. Throughout the water persistence studies there was no significant conversion of terbufos to t.sulfoxide or t.sulfone, or conversion of t.sulfoxide to t.sulfone. The disappearance of the three compounds followed first-order kinetics¹⁹. The half-lives ($t_{1/2}$) of the insecticides in the various treatments, and their respective pH values are shown in Table 2. Since terbufos disappeared as rapidly from distilled water as from either the natural, or sterilized natural water, neither microorganisms nor other naturally occurring compounds in the natural water appeared to influence the rate of terbufos disappearance. pH did not appear to be an important factor in terbufos disappearance since the pH of distilled water was 5.50 (5.90 following treatment) whereas the pH of natural water was 7.93 (8.70 following treatment) and the sterilized natural water was 8.70 (8.75 following treatment). However, pH may have played a role in t.sulfoxide and t.sulfone disappearance in the natural and sterilized natural water where $t_{1/2}$ values were markedly less than for distilled water treatments which were at lower pH values. The alkaline pH of the natural water, taken from a drainage ditch at the Holland Marsh, the analysis of which has been previously reported²¹, is ascribed to the calcareous subsoil below the drainage ditch. The natural water contained some algal material, which must have accumulated calcareous materials which were subsequently released upon autoclaving, thereby increasing the pH to 8.70.

Adsorption Studies. Adsorption data on the four soils for terbufos, t.sulfoxide and t.sulfone were plotted using the modified Freundlich equation³

$$S = K_{MF} Z^N \quad (1)$$

or the linearized log form

$$\log S = \log K_{MF} + N \log Z \quad (2)$$

TABLE 2

First-Order Half-Lives and pH Values for Terbufos, T.sulfoxide and T.sulfone in Natural, Sterilized Natural and Distilled Water at 20°C.

System	t _{1/2} (days)	pH		
		0 Day	1 Wk	16 Wk
Distilled Water		5.50	5.65	
- Terbufos	3.3	5.90	6.05	8.70
- T.sulfoxide	347	6.00	5.90	7.65
- T.sulfone	277	5.90	5.90	7.00
Natural Water		7.93	7.88	
- Terbufos	3.2	8.70	8.80	8.50
- T.sulfoxide	40.8	8.80	8.80	8.50
- T.sulfone	32.1	8.75	8.80	8.50
Sterilized, Natural Water		8.70	8.75	
- Terbufos	3.5	8.75	8.70	8.50
- T.sulfoxide	32.7	8.80	8.75	8.50
- T.sulfone	17.7	8.80	8.80	8.50

where S = moles adsorbate adsorbed/unit weight adsorbent

N = Freundlich slope

Z = mole fraction of adsorbate in solution at equilibrium

K_{MF} = mathematical regression constant having units of moles g⁻¹

In the mole fraction format, the K value (now K_{MF}) loses any physical interpretation since the concentration (mole fraction) at which it would be evaluated (log Z=0, Z=-1 analogous to C=1 when log C=0 in the log Freundlich equation) is far above the solubility limit of any pesticidal compound in aqueous solution. However, this format allows all pesticide adsorption data to be plotted in one consistent set of units - moles.

Relative adsorption comparisons of compounds can be made at any appropriate mole fraction value, and is indicated by "S_{log Z}". This is interpreted as the amount of adsorption, S, that occurs at an equilibrium mole fraction value of log Z. A log Z value of -7 would translate into an equilibrium concentration value in the range of 1.1 to 1.7 g ml⁻¹ for

TABLE 3

Solubility Values, Octanol-Water Partitioning Coefficients and Adsorption Parameters in
Four Soils for Terbufos, T.sulfoxide and T.sulfone (at 20°C)

Adsorbent	Terbufos			T.sulfoxide			T.sulfone		
	N	K_{MF}^*	S_{-7}^*	N	K_{MF}^*	S_{-7}^*	N	K_{MF}^*	S_{-7}^*
			$\times 10^{-8}$		$\times 10^{-3}$	$\times 10^{-8}$		$\times 10^{-3}$	$\times 10^{-8}$
Plainfield sand	1.071	2.793	8.893	0.794	6.132	1.700	0.786	3.619	1.139
Big Creek sediment	1.014	2.409	19.22	0.909	14.81	0.642	0.794	3.962	1.096
Bondhead sandy loam	0.971	1.559	24.88	0.887	24.43	1.510	0.783	3.835	1.267
Muck soil	0.966	13.16	227.6	0.853	77.39	8.273	0.940	303.5	7.983
Solubility (g ml ⁻¹)	5.5			$>1.10 \times 10^3$			4.08×10^2		
(mole L ⁻¹)	1.91×10^{-5}			$>3.61 \times 10^{-3}$			1.27×10^{-3}		
Octanol-Water	3.30×10^4			1.64×10^2			3.02×10^2		
<u>Partitioning Coeff.(23°C)</u>									

* units - mole g⁻¹

compounds ranging in molecular weight from 200 to 300, respectively. The $S_{.7}$ values in Table 3 indicate that t. sulfoxide and t. sulfone adsorption is within 50% of each other whereas terbufos adsorbed from 5 to 30 fold more than either t. sulfoxide or t. sulfone, depending on the soil type.

Although the $S_{.7}$ values for the three compounds varied considerably among the four soils, there appeared to be a relationship with organic matter (O.M.) content. Regression equations of $S_{.7}$ values on percent O.M. were derived for each compound in the four soils. The correlation coefficient for terbufos was highly significant $r \geq 0.99$, 0.01 level, 2 degrees of freedom) indicating a close correspondence between O.M. content and adsorption. However, there was no significant relationship between O.M. content and adsorption of the more water-soluble t. sulfoxide or t. sulfone.

Desorption Studies. Four desorption cycles for t. sulfoxide and t. sulfone were completed on the four soils to determine the relative removal rates (Figure 1). Terbufos was not included in this study because of its tendency to degrade in distilled water at 20% of the amount remaining per day (Table 2), which would lead to unacceptable accuracy over the more than two day period for a desorption experiment. This did not prove to be a problem in the adsorption experiments (lasting <1 day) where more than 93-95% of the initially added terbufos could be accounted for. Both t.sulfoxide and t.sulfone exhibited similar desorption tendencies in all four soils, which is consistent with their similar adsorption behavior shown in Table 3. After four desorption cycles, neither t. sulfoxide nor t. sulfone remained on any of the soils.

Mobility Studies. Most of the t.sulfoxide and t.sulfone was eluted from the mineral soils in the first few fractions (Table 4), consistent with their behavior in the adsorption and desorption studies. The amount of t.sulfoxide and t.sulfone that could be accounted for at the end of the experiment (sum of 10 fractions amount remaining in leached sample) was markedly less in the Big Creek sediment than in the other three soils for some unknown reason. Although terbufos is adsorbed to a greater extent in soils and is less stable than

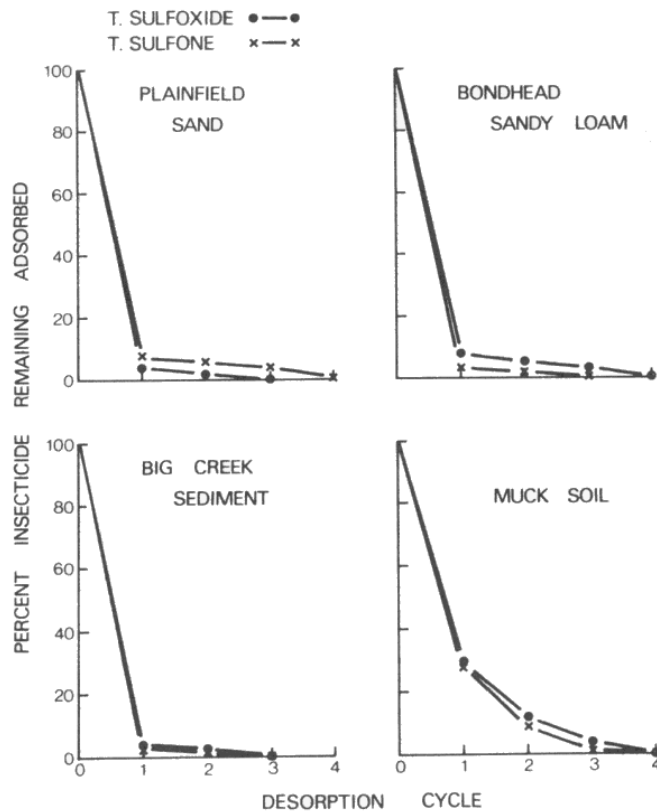


FIGURE 1
Desorption of terbufos sulfoxide and terbufos sulfone from four soils.

the sulfoxide and sulfone, more than 60% of the added terbufos was eluted from the three mineral soils. The total amount of terbufos that could be accounted for at the end of the leaching experiments was considerably less than for t. sulfoxide and t. sulfone in their respective soil systems.

The Mobility Factors (M.F.) described previously^{16,22}, are a means of quantitatively ranking the tendencies of compounds to be leached, where a value of 10 signifies complete removal in the first fraction, and zero signifies no removal whatsoever (Table 4). As might be expected, terbufos had lower M.F. values than did either t. sulfoxide or t. sulfone in their respective soils.

TABLE 4

Mobility of Terbufos, T.sulfoxide and T.sulfone in Four Soils

Soil	Percent (of applied) Leached in Fraction										Total Percent 10 Fractions	Percent left in Sample	Mobility Factor
	1	2	3	4	5	6	7	8	9	10			
<u>Terbufos</u>													
P.S.	70.89	4.84	2.71	1.13	0.84	0.31	0.12	0.02	-	-	80.85	3.72	7.89
B.C.S.	52.93	8.16	3.49	1.45	1.09	0.20	0.15	0.10	0.08	0.06	67.71	-	6.49
B.S.L.	28.07	11.21	6.49	4.04	3.59	2.39	1.89	1.42	0.95	0.88	60.90	0.92	5.10
Muck soil	-	0.60	0.68	0.80	0.64	0.77	1.00	1.53	1.96	2.54	10.50	42.60	0.39
<u>Terbufos sulfoxide</u>													
P.S.	98.42	0.80	0.60	0.73	-	-	-	-	-	-	100.60	-	10.0
B.C.S.	76.96	1.80	1.45	0.28	0.15	0.37	-	-	-	-	81.00	-	8.02
B.S.L.	92.46	2.19	0.76	0.49	0.40	0.40	-	-	-	-	97.57	-	9.58
Muck soil	5.69	6.96	8.62	7.04	5.58	5.20	4.95	4.77	4.40	4.55	57.75	35.20	3.45
<u>Terbufos sulfone</u>													
P.S.	86.60	6.40	2.00	1.30	0.40	0.30	-	-	-	-	97.00	-	9.53
B.C.S.	88.34	0.52	0.06	-	-	-	-	-	-	-	88.93	2.60	8.89
B.S.L.	86.78	6.00	1.87	0.86	0.61	0.24	0.12	0.06	-	-	96.30	-	9.48
Muck soil	6.47	8.26	6.52	6.17	5.64	4.58	4.48	3.93	3.84	3.64	53.52	46.05	3.32

P.S. = Plainfield sand;

B.C.S. = Big Creek sediment,

B.S.L. = Bondhead sandy loam.

Non-detectable <0.02%.

Because of the aforementioned low recovery in the Big Creek sediment systems, the M.F. values may be anomalously low for this soil.

In terms of practical application, this leaching experiment should be regarded as a "worst-case Rainfall Scenario," which tests the ability of a rapid-moving water front to remove insecticides from a small cylindrical plug of treated soil. With this experimental set-up, no consideration was given to re-adsorption and subsequent equilibration of the chemical at lower levels in a simulated soil profile.

Octanol-Water Partitioning Studies. Both the water solubility values, determined previously⁵, at 20°C and the octanol-water partitioning coefficients (K_{OW}) are shown in Table 3. Further efforts at purifying the octanol or water reagents, beyond that already noted, did not produce different results from those shown. A series of K_{OW} determinations for a number of other insecticides, for which published K_{OW} values are available, was used to standardize the technique. Because of the relative instability of terbufos in water, the equilibration time was shortened to 1.5 hr before extraction. A fourth extraction was necessary for the terbufos K_{OW} determination because the concentration in the water layer decreased sharply following the first equilibration cycle, thereby greatly increasing the K_{OW} value.

DISCUSSION

The S_7 values (Table 3) show that terbufos (solubility = 5.5 g ml⁻¹) adsorbed to a much greater extent than the more soluble t. sulfoxide (>1100 g ml⁻¹) or t.sulfone (408 g ml⁻¹). Despite the fact that t. sulfoxide is approximately three times more soluble than t. sulfone, t. sulfoxide adsorbed more strongly than t. sulfone in three of the four soils (excepting Big Creek sediment). Differences between the solubility of compounds often appear to be of diminishing importance in predicting their relative adsorption when solubility values exceed the 50-100 µg ml⁻¹ region. In an earlier study¹⁶ where the solubility of fensulfothion was 2000 g ml⁻¹ and its sulfone was only 74.6 g ml⁻¹, there was again no consistent large difference in their adsorption tendencies in the same three mineral soils. In all four soils, fensulfothion

sulfide¹⁶, adsorbed to a greater extent than terbufos. In three of the soils (excepting Plainfield sand) fensulfothion and f.sulfone both adsorbed to a greater extent than t.sulfoxide or t.sulfone.

The K_{OW} value obtained for terbufos (Table 3), 33000, is at variance with Metcalf and Lu's value of 167²³ or Felsot and Dahm's value of 4796¹⁰. The initial equilibration of the octanol-water mixture in this study produced a K_{OW} value of 8600 for terbufos, which in three successive extractions (in triplicate) shifted to the above-reported 33,000. This large increase in K_{OW} in the second extraction may be attributed to a more water-soluble impurity¹³ which was removed in the first extraction. The disagreement in reported values with Felsot and Dahm¹⁰ may be due to differences in experimental technique. In the present study, the reagent-grade octanol was purified before use, the two solvents were mutually saturated, a larger aqueous volume was used (40 ml vs 10 ml) and the centrifugation intensity (34,800 x g) and duration (1 hr) were significantly different from Felsot and Dahm's work where they centrifuged at 429 x g for 10 min. We found that centrifugation periods less than 0.5 hr (at 34,800 g) produced inconsistent results. It is well known that an inverse relationship exists between log K_{OW} values of compounds and their respective log water solubilities⁹, and that the theoretical slope for this regression is $-1.0^{1,8,15}$. In this study, the slope value for the three terbufos analogs was -1.04, using molar solubility values.

As with the earlier studies with fensulfothion and its related compounds¹⁶, there was good agreement among the adsorption, desorption and mobility data. In all cases terbufos and its sulfoxide and sulfone showed a greater tendency to be leached and/or desorbed from the four soils than did the respective fensulfothion counterparts.

The desorption data both in this study, and in a previous study¹⁶ were generated using samples treated by the spiking method of Sharom et al.²² in order that the data be comparable. It has been recently demonstrated that desorption data derived by this technique can not be directly compared to data obtained by the conventional treatment method (adsorption from aqueous solution)⁶. Consequently, these Freundlich desorption parameters are not shown. However, a Freundlich plot of these data does show irreversibility or hysteresis effects ($N_{Ads} > N_{Des.}$) common to many insecticidal compounds

in soil^{2,20}. The desorption isotherms of Felsot and Dahm¹⁰ for terbufos and several other insecticides show essentially no hysteresis effects ($N_{\text{Ads.}} > N_{\text{Des.}}$) and in fact show several instances where $N_{\text{Ads.}} > N_{\text{Des.}}$. This anomaly occurred because they incorrectly joined a series of first-desorption cycle points to create their desorption isotherm. The initial adsorption point is always unique to each desorption isotherm, and only successive desorptions from this point constitute a desorption isotherm. If Felsot and Dahm had used the same procedure to plot their desorption isotherms as Peck et al.²⁰, their desorption isotherms would have exhibited similar hysteresis effects $N_{\text{Ads.}} > N_{\text{Des.}}$.

Persistence studies in aqueous media showed no evidence of any interconversion among terbufos and its analogs. A similar result was observed in the respective aqueous systems for fensulfothion and its analogs¹⁶. However, in soil, both the terbufos series⁶ and the fensulfothion series^{16,18} showed oxidation of the sulfide to the sulfoxide and subsequently to the sulfone. The disappearance of terbufos, t.sulfoxide and t.sulfone appeared to follow first-order kinetics in water. There was no indication that microorganisms played a role in the disappearance of terbufos or its analogs in any of the aqueous systems.

Results of this and earlier studies suggest that following soil application, terbufos would be adsorbed to a greater extent than fensulfothion (being more comparable to f.sulfide¹⁶) but would degrade in soil at about the same rate as fensulfothion ($t_{1/2} = 1 \text{ wk}$)^{7,18}. However, terbufos appears to be somewhat volatile in soil, as judged by its fumigant action, and therefore would probably be less persistent in soil than fensulfothion⁷. In muck soils, the volatility of terbufos would probably be considerably less than in mineral soils, leaving more terbufos available for conversion to the more stable t.sulfoxide and eventually to t.sulfone, both of which exhibit insecticidal activity, albeit considerably less than that of terbufos in soil⁷. Because of the reasonably strong adsorption by muck soil (relative to mineral soil, Table 3), both t.sulfoxide and t.sulfone are somewhat restricted in their mobility (Table 4) and would not pose a significant environmental hazard. In mineral soils, terbufos would probably not persist long enough for significant conversion to t.sulfoxide or t.sulfone. If any terbufos happened to reach groundwater (unlikely because of its persistence and adsorption behavior), it would be an unlikely pollution source because of its short half-life in water of

about 3 days. Terbufos sulfoxide and t.sulfone in mineral soil are more subject to leaching and are also more stable in groundwater than terbufos, but shouldn't pose any environmental threat because of 1) minimal amounts likely to be formed by conversion from terbufos and 2) their low toxicity.

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