Sorption Behavior of *s*-Triazine and Thiocarbamate Herbicides on Soils

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ABSTRACT

The soil-water partitioning coefficient (K_d) is an important parameter for predicting the transport of herbicides in soils. We studied the sorption behavior of two *s*-triazines [atrazine (2-chloro-4-ethylamino-6-isopropylamino-1,3,5 triazine) and prometon (2,4-bis(isopropylamino)-6-methoxy-*s*-triazine)] and two thiocarbamates [EPTC (*s*-ethyl dipropylthiocarbamate) and triallate (*s*-(2,3,3-trichloroallyl)diisopropyl-thiocarbamate)] on two soils [Hanford sandy loam (HSL) (coarse-loamy, mixed, nonacid, thermic Typic Xerorthents) and Tujunga loamy sand (TLS) (mixed, thermic Typic Xeropsamments)] with varying organic matter content and soil texture. The K_d-values were determined with a mass balance equilibrium batch technique in which the solution as well as the sorbed phase concentration was directly measured. Two grams of soil were equilibrated with 20 mL of aqueous herbicide solution for 24 h at 25 ± 1 °C. Four concentrations of each herbicide at one-half, one-fourth, and one-sixth, and one-eighth of their respective aqueous solubilities were selected for the experiments. Sorption constants were calculated with both linear and nonlinear Freundlich isotherms. The exponents $1/n$ of the Freundlich isotherm varied between 0.767 and 0.984, thus indicating considerable nonlinearity in some of the isotherms. The K_{oc}-values (soil-water partitioning constants based on the organic C fraction of a soil) were calculated as the ratio of the measured K_d-values and the soil organic fraction of the HSL and TLS soils. Using the mass balance equilibrium method, the average K_{oc}-values of atrazine, EPTC, prometon, and triallate for the two soils were found to be 54, 196, 76, and 2838 m^3/Mg, or 0.42, 0.50, 0.21, and 0.88 times those estimated from measured soil solution concentrations only. Since the latter values are much closer to reported literature values (most of which were determined from measured solution concentrations only), we believe that the variability and ambiguity of current literature K_{oc}-values may be due largely to inaccurate determination of K_d-values. We also estimated the K_{oc}-values from basic soil physical and chemical properties by using previously reported predictive equations.

AGRICULTURE PRODUCTION has greatly benefited from the use of herbicides. Unfortunately, the heavy reliance on herbicides also raises the question of how to protect the long-term quality of our soil and water resources. As detailed screening of each herbicide under laboratory or field conditions is not feasible, several mathematical simulation approaches have been proposed for predicting the behavior of these organics in the environment (e.g., Lindstrom et al., 1968; van Genuchten et al., 1974, 1977; Knisel, 1980; Farmer et al., 1980; Jury et al., 1983a, 1987; Donigian and Rao, 1986). The calibration, validation, and best use of these predictive approaches require accurate values for benchmark properties of the chemicals and their degradation products.

Sorption and degradation are the two most important processes affecting the fate and behavior of pesticides in soil. The degree of sorption from the solution onto the solid phase of the soil may be described by a soil-water partitioning coefficient (K_d). Previous studies have shown that for most nonionic chemicals, K_d is determined primarily by the soil organic C fraction (F_{oc}) (e.g., Bailey and White, 1972; Rao and Davidson, 1980; Green and Karickhoff, 1986). The K_{oc}-values can provide a correlation between K_d and F_{oc}, and thus make it possible to specify adsorption properties of a herbicide largely independent of soil type. The K_{oc}-values are highly correlated with water solubility, water-octanol partition ratios (K_{ow}), bioconcentration factors, equilibration times for pesticide-soil-water-systems, and selected soil properties including pH, particle-size distribution, and surface area (Rao and Davidson, 1980; Green and Karickhoff, 1986). Several studies (e.g., Jury et al., 1983a; Villeneuve et al., 1988) have directly incorporated K_{oc}-values into mathematical models for predicting the fate and transport of organic chemicals in the environment.

Although a large number of laboratory methods exists for measuring sorption from solution (Green et al., 1980), the most commonly used technique is the batch equilibration method. Most K_d- and K_{oc}-values currently available in the literature are derived from batch equilibrium experiments in which the adsorbed phase concentration is not directly measured but indirectly calculated from changing solution phase concentration. Such indirect solution methods do not allow for an independent verification of the mass balance of the chemical during the experimental procedure. Variable soil properties may also have resulted in significant variations in reported K_{oc}-values (Hamaker and Thompson, 1972; Rao and Davidson, 1980; Green and Karickhoff, 1986). The reported variability in K_{oc}-estimates cast considerable uncertainty as to the applicability of simulation results obtained with theoretical transport models. An alternative and

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more accurate technique for measuring sorption is to use a mass balance method in which both the solution and sorbed phase concentrations are measured independently (McCall et al., 1981). This paper reports comparisons of K_{oc}-values of four commonly used herbicides (atrazine, EPTC, prometon, and triallate) for two different California soils as determined with both the mass balance method and the more conventional solution method. Hence, one major objective of this study was to determine the accuracy of the mass balance method as compared to the solution method.

MATERIALS AND METHODS

Herbicides

Four commonly used nonionic herbicides, viz, atrazine, EPTC, prometon, and triallate were selected for our study. These organics are preemergence herbicides commonly used to control weeds in many crops. Analytical standards of the chemicals were obtained from the USEPA (Research Triangle Park, NC). Selected properties of the four herbicides are given in Table 1.

Soils

Two California soils, a Hanford sandy loam (HSL) from the Moreno Experimental Farm of the University of California, Riverside, and a Tujunga loamy sand (TLS) from Fontana, were selected for the experiments. They represent the soils of two research farms of the University of California, Riverside. Bulk soil surface (0–15 cm) samples were air-dried, passed through a 2-mm sieve, and analyzed for their basic physical and chemical properties. Standard methods were employed to determine the pH (1:1, water/soil), total organic C (dry combustion), cation exchange capacity (CEC) (1:1 Na saturation), and particle size distribution (hydrometer); results are listed in Table 2.

Batch Equilibrium Studies

Aqueous solutions of each herbicide were prepared from hexane/acetone solutions of the analytical grade herbicides. The initial concentrations were approximately one-half, one-fourth, one-sixth, and one-eight of the aqueous solubilities of each herbicide. Equivalent amounts of the analytical grade stock solution for each concentration were deposited on the inner walls of 1-L volumetric flasks, after which the solvents were completely evaporated with a gentle stream of dry N_2. Next, the deposited pesticides were solubilized in 0.01 *M* $CaCl_2$ aqueous solutions. While some previous workers have used different soil/water ratios, a majority preferred 1:10 ratios for their sorption studies. Hence, we also selected the 1:10 ratio. Two grams of soil and 20 mL of the aqueous solution were equilibrated in a 35-mL Teflon test tube by using an end-over-end electrical shaker at 40 rpm for 24 h at 25 ± 1 °C. All treatments were replicated four times. Several water-soil blanks were used. After equilibration, the tubes were centrifuged in a thermostable high-speed centrifuge at 14 000 rpm (23 500 × *g*) for 10 min at 25 ± 1 °C. The herbicide concentrations in soil and supernatant liquid phases at equilibrium were determined using methods described below.

Table 1. Selected properties (at 25 °C) of the four herbicides used in this study.

Herbicide	Mole. wt.	Vapor pressure	Vapor density	Solubility	K_H†	Half-Life (in soil)
		Pa	mg/L			d
Atrazine‡	215.7	9.0E–05§	8.0E–06	33	2.5E–07§	71
EPTC‡	189.3	2.8	2.2E–01	370	5.9E–04	30
Prometon#	225.3	8.3E–04	7.5E–05	750	1.0E–07	–
Triallate‡	304.7	2.6E–02	3.2E–03	4	7.9E–04	100

† Henry's law constant.

‡ Jury et al. (1983b).

§ 1.0E–05 = 1.0 × 10^{-5}.

Spencer et al. (1988).

Table 2. Some properties of the soils used in the adsorption studies.

Soil type	Organic carbon	pH (1:1)	CEC at 25 °C	Clay <2 μm	Silt 2–50 μm	Sand 50 μm–2 mm
	%		cmol/kg	%		
Hanford sandy loam (HSL)	0.43	6.05	5.95	7.1	25.8	67.1
Tujunga loamy sand (TLS)	0.33	6.30	0.45	4.5	13.5	82.0

Extraction

Chemicals in the liquid phase were extracted by a liquid-liquid partitioning technique using *n*-hexane for atrazine, EPTC, and triallate, and dichloromethane for prometon. The supernatant solutions from the tubes were transferred into 125 mL separatory funnels and diluted with 20 mL of a 5% (w/v) sodium carbonate aqueous solution. Herbicides were then extracted four times with 25 mL solvent by vigorous shaking for 30 s each time. The four solvent batches were combined and transferred through a 5-cm anhydrous sodium sulfate column to a wide-mouth, round-bottom flash evaporator (250 mL) flask. The hexane extracts containing herbicide residues of atrazine, EPTC, and triallate were concentrated to 25 mL, whereas the dichloromethane extracts of prometon were dried completely and solubilized in hexane. The final volume of each extract was 25 mL.

The soil samples were mixed with washed and dried silica sand, and extracted by Soxhlet extraction using 200 mL hexane + acetone (1:1, v/v) for 4 h. The extracts were passed through a 5-cm anhydrous sodium sulfate column and concentrated on a rotary flash evaporator. Adsorbed concentrations were corrected for the amount of pesticides in the residual aqueous solution remaining in the soil.

Analysis

Herbicide concentrations in the extracts were analyzed using a Varian 3700 gas liquid chromatograph (GLC) equipped with NP and ^{63}Ni electron capture detectors (Varian Instruments, Sunnyvale, CA). The GLC conditions for the analysis of different compounds are given in Table 3. With the methodology adopted here, the recovery of the compounds separately from the fortified soil and water samples varied be-

Table 3. Gas liquid chromatograph conditions for analyses of four herbicides.

Herbicide	Temperature, °C			Attenuation × 10^{-12}	Gas flow, mL/min			Retention time, min
	Injection port	Column†	Detector (NP)‡		H_2	N_2	Air	
Atrazine	200	150	300	8	18	20	20	1.5
EPTC	150	110	300§	2	–	20	–	2.43
Prometon	200	180	300	16	18	20	20	1.95
Triallate	180	155	250	2	18	20	20	2.03

† Columns: 0.91 m × 2 mm i.d. Ultrabond 20M on carbowax 0.125 to 0.149 mm and for EPTC DB-Wax with same material (Megabore) for all others (Alltech Assoc., Los Altos, CA).

‡ Nitrogen phosphorus.

§ ^{63}Ni electron capture detector.

tween 97 and 102%. However, from the soil-water-herbicide batch experiments, the mass of herbicides recovered was found to vary between 90 and 97%.

Sorption Isotherms

Herbicide sorption isotherms were calculated using the Freundlich equation

$$C_s = KC_w^{1/n} \qquad [1]$$

where C_s is the sorbed concentration (mg/kg), C_w is the equilibrium solution concentration (µg/mL), and K and $1/n$ are the Freundlich equilibrium constants. The nonlinear least-squares optimization computer program developed by Zhang et al. (1987) was used to estimate the model parameters. We also described pesticide sorption in terms of the simpler linearized relationship

$$C_s = K_dC_w \qquad [2]$$

where K_d is the linear partitioning coefficient.

RESULTS AND DISCUSSION

Table 4 summarizes the equilibrium distribution of the four herbicides in the adsorbed and solution phases. The amounts of herbicide applied and percentile recovered, are also included. The K_{oc}-values were calculated using the organic C fraction (F_{oc}) of each soil and the Freundlich K-values, as determined with the mass balance and the solution concentration methods. Optimized values of K and $1/n$ (Eq. [1]) and K_d-values (Eq. [2]) in the sorption isotherms for different herbicide-soil combinations are listed in Table 5. Note that in most cases the calculated K_d-values are significantly less than those calculated with the Freundlich Eq. [1]. This is because Eq. [2] does not consider nonlinearity in the sorption isotherm. As previously shown by Rao and Davidson (1982), K_d-values obtained by applying Eq. [2] may lead to large errors when n deviates significantly from unity.

As previously discussed, most earlier workers calculated K or K_d on the basis of solution concentrations (C_w) only. This approach assumes that the remainder of the pesticide is adsorbed as C_s, and ignores degradation, volatilization, or other processes causing disappearance or nonrecovery of herbicides from the measurement system. For example, of 29.7 µg atrazine applied to the HSL soil (Table 4), only 28.68 µg was recovered using the mass balance method. In the solution method, the difference of 1.02 µg is immediately contributed to adsorption, thus increasing the sorbed phase concentration by 0.51 mg/kg, and ultimately overpredicting by 2.19 times the adsorption coefficient as compared to the mass balance method. For comparison, we have included in Table 5 also the Freundlich equation parameters calculated on the basis of solution phase concentrations only. The same trend of adsorption behavior of the eight herbicide-soil combinations occurred with both methods. The data in Table 5, however, indicate that the Freundlich K-values calculated with the solution method are in all cases

Table 4. Equilibrium distribution of herbicides between adsorbed and solution phases.

						K_{oc}‡	
Herbicide	Soil†	Adsorbed concentration	Solution concentration	Mass of herbicide applied	Average recovered	Mass balance	Solution method
		µg/g	µg/mL	µg	%	m³/Mg	
Atrazine	HSL	0.34	1.40	29.7			
		0.65	2.94	62.0			
		1.25	6.76	142.3			
		1.61	10.38	220.0	97.3§	63	143
	TLS	0.37	2.93	61.5			
		0.47	4.27	90.3			
		0.73	6.53	136.4			
		0.93	9.12	190.4	96.6	45	112
EPTC	HSL	19.71	28.90	686.8			
		26.91	41.86	990.4			
		66.18	111.30	2620.8			
		136.10	189.60	4514.7	90.1	148	424
	TLS	11.03	23.50	546.6			
		19.05	44.57	1032.5			
		25.17	80.40	1843.1			
		41.63	100.40	2323.2	90.0	242	572
Prometon	HSL	11.39	52.06	1138.7			
		20.09	105.00	2272.0			
		31.97	176.90	3892.2			
		39.74	215.00	4738.0	91.8	84	395
	TLS	10.01	51.25	1158.5			
		18.84	102.50	2329.0			
		31.97	175.60	3920.5			
		39.91	213.60	4767.3	91.9	67	340
Triallate	HSL	1.68	0.13	6.2			
		3.83	0.29	14.1			
		5.87	0.43	21.4			
		8.23	0.70	32.0	94.9	2832	3397
	TLS	1.98	0.13	7.3			
		3.21	0.27	11.9			
		5.79	0.48	24.0			
		7.30	0.74	32.9	91.4	2843	3072

† HSL = Hanford sandy loam; TLS = Tujunga loamy sand.

‡ Calculated using the F_{oc} of each soil and the Freundlich K-values determined with the mass balance and solution methods.

§ Each value is the mean of four replicates and four concentrations.

substantially higher than the mass-balance computed values.

The observed differences in sorption behavior of the four herbicides were expected because of their different mode of adsorption on soil substrates (Mortland and Meggitt, 1966; Calvet, 1980; Beste and Humburg, 1983; Gamble et al., 1986; Madhun et al., 1986). Irrespective of soil or calculation method, adsorption of triallate on the two soils was always greatest, followed in order by EPTC, prometon, and atrazine. Atrazine, prometon, and triallate adsorption on HSL was higher than on TLS (Table 4) because of slightly higher organic matter and clay contents (Borggaard and Streibig, 1988; Ramen et al., 1989). EPTC showed little difference in adsorption on the two soils, but its adsorption on TLS was slightly more nonlinear ($1/n$ = 0.826) than on HSL ($1/n$ = 0.984). Although the organic C contents of the two soils are not much different, the type of the organic matter (OM) may also have played a role. In addition, the higher clay content, higher CEC, and different OM-mineral relations of HSL may have contributed somewhat to the higher adsorption coefficients.

The K_{oc}-values for each herbicide, as calculated with different methods, are summarized in Table 6. Experimental results are compared with available literature values (some of which were estimated from previously measured K_d-values and soil organic C content) along with K_{oc}-values calculated from basic soil and herbicide properties using several equations taken from the literature. Some researchers (e.g., Hamaker and Thompson, 1972) have suggested that one should obtain essentially the same K_{oc}-value for a given pesticide in a variety of soils. This may not always be the case because adsorption of a particular pesticide depends not only on the organic C of the soil, but also the type of organic matter (Ishiwatari, 1969), clay content (McCall et al., 1980), pH of the media (Weber, 1966), CEC (Ghani and Allbrook, 1986), and soil texture (Valverde-Garcia et al., 1988), as well as on the chemical properties of the herbicide itself. Relative differences in K_{oc}-values between the four herbicides were almost the same, irrespective of the method of data calculation. The K_{oc}-values were considerably less (except for triallate using the mass balance method) when calculated using the linear isotherm K_d-values, than when using the nonlinear Freundlich K-values. The solution method (C_w) yielded K_{oc}'s that were 2.4, 2.5, 4.8, and 1.19 (averages of both soils) times those by the mass balance method for atrazine, EPTC, prometon, and triallate, respectively.

The literature values listed in Table 6 vary greatly.

Table 5. Parameters of the linear and nonlinear Freundlich isotherms for different herbicide-soil combinations.

				Freundlich isotherm parameters§					
		Linear equation parameters‡		Mass balance			Solution method		
Herbicide	Soil†	K_d	r^2	K	$1/n$	r^2	K	$1/n$	r^2
Atrazine	HSL	0.08	0.977	0.27	0.789	0.998	0.61	0.921	0.999
	TLS	0.09	0.963	0.15	0.833	0.996	0.37	1.297	0.765#
EPTC	HSL	0.47	0.631#	0.72	0.984	0.995	1.81	1.004	0.999
	TLS	0.36	0.907	0.80	0.826	0.971	1.90	0.951	0.998
Prometon	HSL	0.15	0.939	0.36	0.872	0.999	1.68	0.918	0.999
	TLS	0.17	0.927	0.22	0.963	0.999	1.13	0.993	0.999
Triallate	HSL	14.49	0.900	12.07	0.941	0.997	13.10	0.943	0.997
	TLS	8.90	0.976	9.43	0.767	0.994	11.27	0.783	0.995

† HSL = Hanford sandy loam; TLS = Tujunga loamy sand.
‡ Calculated with linear Eq. [2] from C_s/C_w ratios using the mass-balance method.
§ Calculated with the nonlinear Eq. [1].
# All values except these were significant at the 95% confidence level.

Table 6. Experimental K_{oc}-values (m³/Mg) of four herbicides compared with literature values and values estimated from physico-chemical properties.

	Based on this study								
		Freundlich K			Estimated from equations‡				
Herbicide	Linear K_d	Mass balance	Solution method	Literature values†	A	B	C	D	E
Atrazine	23	54	128	62(4), 130(5), 11(6), 102(7), 258(10), 122(11), 102–163(8)	638	1526	1888	192	1599
EPTC	110	196	498	48–136(2), 240, and 238(8)	169	393	332		638
Prometon	45	76	358	408(3), 54(4), 197(5), 524(9), 74, and 350 (8)	115	265	200	410	488
Triallate	3041	2838	3235	3600(1), 2200(8)	2037	4986	8612		3569

† Sources: (1) Jury et al. 1980; (2) Ekler, 1988; (3) Zhang et al., 1987, (4) Scott and Phillips, 1972; (5) Harris, 1966; (6) Grover and Hance, 1969; (7) Huang et al., 1984; (8) Green and Karickhoff, 1986; (9) Rao and Davidson, 1980; (10) Clay et al., 1988.

‡ (A) $\log K_{oc} = 3.64 - 0.55 (\log s)$ Kenega and Goring, 1980;
(B) $\log K_{om} = -0.561 \log s + 3.8$ Gerstl and Mingelgrin (1984);
(C) $\log K_{ow} = -0.699 \log s + 4.42$ Gerstl and Mingelgrin (1984);
$\log K_{oc} = 1.029 \log K_{ow} - 0.18$ Rao and Davidson (1980);
(D) $K_d = \text{mSS} = \text{m}(100\% \text{ OC}) + 2(\% \text{ clay}) + 0.4(\% \text{ silt}) + 0.005(\% \text{ sand})$ Pionke and DeAngelis (1980);
(E) $\log K_{ow} = -0.699 \log s + 4.42$ Gerstl and Mingelgrin (1984),
$\log K_{oc} = 1.377 + 0.544 (\log K_{ow})$ Kenega and Goring (1980)

The mass balance K_{oc}'s fall into the lower range and the C_w-based K_{oc}'s in the upper range of these values. Hence, some of the variability in available literature values likely results from the method by which K_d's and K_{oc}'s are calculated. Other factors responsible for this large deviation could be the use of different experimental methodologies, including the use of different soil-solution ratios, equilibration times, initial concentration ranges of the compounds, and imposed pH's of the media (Hamaker and Thompson, 1972).

The K_{oc}-values indirectly calculated with different equations (Table 6) predict varying adsorption behavior of the four herbicides. Particularly, all equations predict much stronger adsorption of atrazine than indicated by our experiments, or reported literature values. Also, the order of increasing adsorption changes from atrazine–prometon–EPTC–triallate to prometon–EPTC–atrazine–triallate with these predictive equations. Equation A gives K_{oc}-predictions that are closer to all our experimental values, except for atrazine.

SUMMARY AND CONCLUSIONS

Adsorption coefficients for atrazine, EPTC, prometon, and triallate were measured by the batch equilibrium method on two soils varying in organic content and soil texture. The data indicate that the mass balance approach, wherein concentrations of pesticides are determined on the adsorbent as well as in the equilibrium solution, is necessary to obtain accurate values with the batch method. Adsorption coefficients calculated using only equilibrium solution concentrations, and assuming that any unrecovered pesticides were adsorbed on the soil, yielded uncertain results. Freundlich adsorption coefficients calculated using solution concentrations only were much higher than the mass balance computed values. This indicates that much of the unaccounted pesticide was probably not adsorbed on the soil, but might have been lost by degradation or other means. Because the reported literature values are closer to those derived from measured solution concentrations only, we believe that much of the variability of current K_{oc}-literature values may have been caused by inaccurate determinations of K_d-values using solution concentrations only. Higher K_d-values obtained with the solution method will lead to an underestimation of the rates of pesticide transport in soils, and overestimate erosion losses. In order to obtain accurate adsorption coefficients by the equilibrium batch technique, we recommend measuring concentrations in the adsorbed phase as well as in the solution phase at equilibrium. This is especially important for the relatively high water/soil ratios as used in our study. Errors associated with the solution method should be less at relatively low water/soil ratios.

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