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## Partitioning Behavior and the Mobility of Chlordane in Groundwater

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■ Batch solubility experiments showed an increase in chlordane solubility from 32  $\mu\text{g/L}$  in pure (18 M $\Omega$  cm) water to over 400  $\mu\text{g/L}$  in the presence of humic substances extracted from geologic material collected near a hazardous waste site in Memphis, TN. Solubility was also enhanced to 52  $\mu\text{g/L}$  in groundwater collected upgradient of the site (total organic carbon, TOC = 1.8 mg/L) and to 146  $\mu\text{g/L}$  in downgradient groundwater (TOC = 34 mg/L). Batch sorption studies demonstrated the tenacious binding of chlordane to the solid phase (distribution coefficients,  $K_d$  = 18-220 mL/g). Column studies showed chlordane mobility to be significantly enhanced by the presence of humic substances. Mean chlordane breakthrough occurred at 20 and 82 pore volumes when columns of geologic material collected near the site were eluted with humic solutions of 25 and 2.5 mg/L as dissolved organic carbon, respectively.

### Introduction

The environmental fate of a compound is governed by the properties of the chemical, its surrounding medium, and their interaction. Chlordane, an organochlorine insecticide (1,2,4,5,6,7,8,8-octachloro-2,3,3a,4,7,7a hexahydro-4,7-methanoindane), exhibits low aqueous solubility (reported values range from 9 to 56  $\mu\text{g/L}$ ), high octanol-water partitioning (log  $K_{ow}$  = 5.58), and persistence in the environment with a reported half-life of about 10 years (1, 2).

In the subsurface environment, mobilization is influenced by the distribution of chlordane between the aqueous phase (mobile) and the solid phase (immobile).

Table I. Chlordane Adsorption to Laboratory Materials<sup>a</sup>

material	% recovered in solution	% adsorbed
unsilanized glass	78	22
silanized glass	42	58
Teflon	54	46
polypropylene	13	87

<sup>a</sup> Solutions spiked with 23.5  $\mu\text{g/L}$  [<sup>14</sup>C]chlordane.

This distribution is made more complex by the presence of humic substances in the aqueous phase and organic matter associated with the solid material. Studies conducted with other hydrophobic compounds have shown aqueous solubility to be enhanced by the presence of humic substances (3-6), implying an increase in effective concentration in the mobile phase. However, increased sorption (and hence immobilization) with increasing organic content of the solid phase has also been well documented (7-9).

A number of workers have developed linear relations between aqueous solubility, soil-water distribution, octanol-water partitioning, and bioconcentration for organic compounds (10-12). However, as noted by Kenaga and Goring (12), calculated values can vary from experimentally determined values by as much as 1 or 2 orders of magnitude. A more accurate assessment of environmental fate requires values to be site specific.

North Hollywood Dump is a closed municipal/industrial landfill located on the alluvial plain and abandoned channels of the Wolf River in Memphis, TN. Elevated levels of chlordane have been found in soils and sediments (>100 mg/kg) and riverine fish (25 mg/kg) in the immediate vicinity of the site (13). The following study was undertaken to assess the use of laboratory batch solubility and sorption experiments in combination with column studies to examine chlordane partitioning and mobilization

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Table II. Distribution Coefficients and Properties of Geologic Material from North Hollywood Dump

sample <sup>a</sup>	collection depth (ft)	$K_d$ (mL/g)	$\log K_{oc}$ (mL/g)	sand (%)	silt (%)	clay (%)	texture	organic carbons (%)
M1	2-3	18	4.30	36	49	15	loam	0.09
2	0-1	23 <sup>b</sup>	4.76	90	6	4	sand	0.04
U2	0-1	28	4.85	90	6	4	sand	0.04
D2	0-1	30	4.88	90	6	4	sand	0.04
M3	3-4	30	4.40	9	75	16	silt-loam	0.12
H3	3-4	53	4.65	9	75	16	silt-loam	0.12
M4	6-7	112	4.45	2	72	26	silt-loam	0.40
M5	7-8	128	4.59	15	72	13	silt-loam	0.33
6	9-10	155 <sup>b</sup>	4.63	3	87	10	silt	0.36
U6	9-10	190	4.72	3	87	10	silt	0.36
D6	9-10	220	4.78	3	87	10	silt	0.36
M7	0-1	25	4.62	96	4	0	sand	0.06
M8	1-2	137	4.55	63	36	1	sandy-loam	0.39
M9	7-8	202	4.51	7	87	7	silt	0.62

<sup>a</sup>The following abbreviations are used: M, Milli-Q water used as the aqueous phase; U, upgradient groundwater used as the aqueous phase; D, downgradient groundwater used as the aqueous phase; H, commercial humic acid added to aqueous phase. <sup>b</sup> $K_d$  measured from a 5% methanol solution.

in groundwater near the site.

### Experimental Section

**Materials.** Uniformly ring-labeled [<sup>14</sup>C]chlordane of specific activity 13.7 mCi/mmol [Velsicol Chemical Co., Chicago (Use of the firm name in this paper is for identification purposes only and does not constitute endorsement by the U.S. Geological Survey.)] was used in all experiments. Although greater recoveries of [<sup>14</sup>C]chlordane were obtained from unsilanized glass (Table I), poly(tetrafluoroethylene), 50-mL screw-cap, centrifuge tubes were used because of their durability in batch solubility and sorption experiments. Aqueous samples were mixed with Ecoscint-A (National Diagnostics), and [<sup>14</sup>C]chlordane concentrations were determined by using a Beckman 7000 liquid scintillation counter.

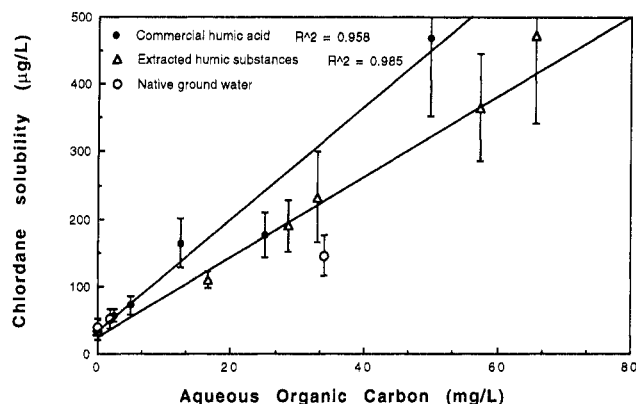
Native geologic materials near North Hollywood Dump were collected with a hand-held 4 cm diameter soil corer at depths of up to 10 ft (Table II). Percentages of sand, silt, and clay were determined for each sample using hydrometer analysis (14), and texture was determined using a textural classification triangle (15). Soil organic carbon was measured by determining total organic carbon following destruction of inorganic carbon with excess 7% sulfurous acid (16). The pretreated soils were combusted at 900 °C in a stream of pure oxygen using a Harvey Instrument Corp. OX400 biological oxidizer. The carbon dioxide generated was trapped in 0.5 N potassium hydroxide, treated with saturated barium chloride, and titrated with 0.25 N hydrochloric acid following the addition of a few drops of phenolphthalein. Percent organic carbon was calculated by the difference between blank and sample titrations. Total organic carbon (TOC) was measured in groundwater collected hydraulically upgradient and downgradient of the landfill (17). Humic substances were isolated from geologic samples by alkaline extraction (0.5 N sodium hydroxide) and filtration through 0.7- and 0.45- $\mu$ m filters, followed by acid precipitation, centrifugation, and freeze-drying of the solid. Commercial humic acid (Aldrich Chemical Co.) was purified in a similar manner. The pH of extracted humic acid solutions was adjusted to 6.5, which is within the pH range of groundwater at the landfill.

**Solubility.** Radiolabeled chlordane was coated to the inside of centrifuge tubes in an amount that would exceed quoted solubilities (1170  $\mu$ g/L if all went into solution). Following slow evaporation of the carrier solvent (methanol), the tubes were filled in triplicate with 40 mL of pu-

rified water (from a Milli-Q ion-exchange system operated at 18 M $\Omega$  cm resistance) augmented with varying concentrations of humic acid and tightly sealed. Following gentle agitation for 48 h at 24  $\pm$  1 °C, the tubes were centrifuged at 12000g for 30 min at 24  $\pm$  1 °C. A 1-mL aliquot was removed from each tube with a glass pipet and added to 10 mL of Ecoscint-A along with hexane rinses of the pipet.

Similar experiments were run using purified water alone and groundwater-collected upgradient (TOC = 1.8 mg/L) and downgradient (TOC = 34 mg/L) of the landfill.

**Batch Sorption Studies.** Because of low aqueous solubility and significant adsorption of chlordane to container walls, measurement of aqueous-phase concentrations of chlordane in standard batch sorption experiments was not feasible. By conducting experiments in the presence of a cosolvent, the equilibrium concentration in the pure aqueous phase was determined by extrapolation (18, 19). Air-dried geologic material collected near the landfill (Table II) was homogenized and sieved (2-mm mesh sieve), and subsamples were weighed into centrifuge tubes. Triplicate cosolvent solutions (made 0.01 N with respect to calcium chloride) of 0-20% methanol in purified water were added and spiked with 20  $\mu$ L of radiolabeled chlordane in methanol (initial chlordane concentration, 150  $\mu$ g/L). Solid to solution ratios in all sorption experiments were 1:1 by weight. Control samples containing no solid material were also prepared, as were samples with no radiolabeled spike. The tubes were shaken continuously in the dark for 24 h at 24  $\pm$  1 °C prior to centrifugation at 12000g for 30 min at 24  $\pm$  1 °C. Preliminary experiments had shown that 24-h agitation was sufficient for the system to reach equilibrium. A 1-mL aliquot of the supernatant was analyzed as previously described. The excess solution from each tube was decanted, and the solid fraction was shaken from the tubes onto aluminum foil and allowed to slowly air-dry at ambient temperature. A 0.50-0.75-g subsample was combusted at 900 °C; the [<sup>14</sup>C]CO<sub>2</sub> thus generated was trapped (R. J. Harvey Instrument Corp. carbon 14 cocktail) and counted to measure that fraction of chlordane sorbed to the solids. Distribution coefficients ( $K_d$ ) were calculated as the ratio of chlordane activity in the solid (dpm/g) and liquid (dpm/mL) phases. A plot of  $\log K_d$  against the fraction of cosolvent allowed extrapolation to the  $K_d$  value for the aqueous phase. The experiments were repeated with upgradient and downgradient groundwater and with commercial humic acid in the aqueous phase. In addition, sorption isotherms were generated for two samples using a single methanol con-



**Figure 1.** Correlation between chlordane solubility and aqueous organic carbon from different sources (lines are least-squares regressions;  $n = 3$ , bars represent standard deviations).

centration, but varying initial chlordane concentrations.

**Column Studies.** To further explore the distribution of chlordane between mobile and immobile phases, experiments were conducted with columns containing a sand collected near the landfill (Table II, solid 7). The fraction organic carbon of this sand was 0.0006. The effect of humic substances on chlordane mobility was determined by leaching the columns with solutions of different humic acid concentration. Glass columns (6-cm i.d.) were first packed with a plug of glass wool and then uniformly packed with wet sand in 1–2-cm increments to a depth of 15 cm. Pore volumes were determined on each column with blue dextran. [ $^{14}\text{C}$ ]Chlordane in methanol was loaded onto the top of each column. Following slow evaporation of the solvent, a humic acid solution of 25 mg/L as dissolved organic carbon (DOC) was made up in U.S. EPA moderately hard water (20), filtered through a 0.45- $\mu\text{m}$  Millipore filter, and eluted through duplicate columns at a rate of 0.8–1 mL/min. A constant head of 4 cm was maintained on each column by the continuous addition of humic acid solution at 0.8–1 mL/min from feed carboys. Column effluents were sampled twice a day, 1 mL from the total volume collected over each time interval and 1 mL collected directly from the column. The samples were analyzed as described above. At the conclusion of each column run, the column was sectioned into 1-cm vertical increments and each increment was analyzed for its organic carbon content and solid-phase chlordane concentration.

### Results and Discussion

**Solubility.** Chlordane solubility increased linearly with the DOC concentration of the humic samples (Figure 1). With commercial humic acid, solubility was enhanced from 30  $\mu\text{g/L}$  in purified water to 470  $\mu\text{g/L}$  in a humic solution of 50 mg/L as DOC. There was an increase in chlordane solubility to 470 and 370  $\mu\text{g/L}$  in the presence of humic substances extracted from two geologic samples from the site at DOC concentrations of 70 and 60 mg/L, respectively. Also, experiments with groundwater collected up-gradient and down-gradient of the landfill indicated an increase in chlordane solubility to 50 and 150  $\mu\text{g/L}$ , respectively (Figure 1).

Because humic substances contain nonpolar regions or domains within the molecule (21), solubility enhancement has been attributed to the partitioning of the solute into hydrophobic regions of dissolved humic materials (22). Such partitioning into humic substances has been described by a partition coefficient based on DOC,  $K_{\text{doc}}$ , obtained from the slope and intercept of the regression lines in Figure 1 using the relation developed by Chiou et al. (22):

$$S_w^* = S_w K_{\text{doc}} x + S_w \quad (1)$$

where  $S_w^*$  is the effective solubility of the solute in water with a DOC concentration of  $x$  (g/mL) and  $S_w$  is the solubility in pure water. By use of such a relation and the data from Figure 1, a  $\log K_{\text{doc}} = 5.45$  was obtained for chlordane solubility in commercial humic solutions, and a  $\log K_{\text{doc}} = 5.31$  in water-containing humic substances extracted from the site. The effective solubility of chlordane in native groundwater is less than that in water with comparable concentrations of humic substances from commercial sources or derived from extracts of native geologic material. This result may reflect the more hydrophilic nature of humic acids in groundwater (23, 24). Carter and Suffet (25) found that the solubility of DDT in seven different humic materials depended not only on concentration of the humic solutions but also on the source of the humic material. It is well-documented that commercial humic acids are not representative of naturally occurring humic substances (26); data from experiments using commercial material should therefore be interpreted with caution (4).

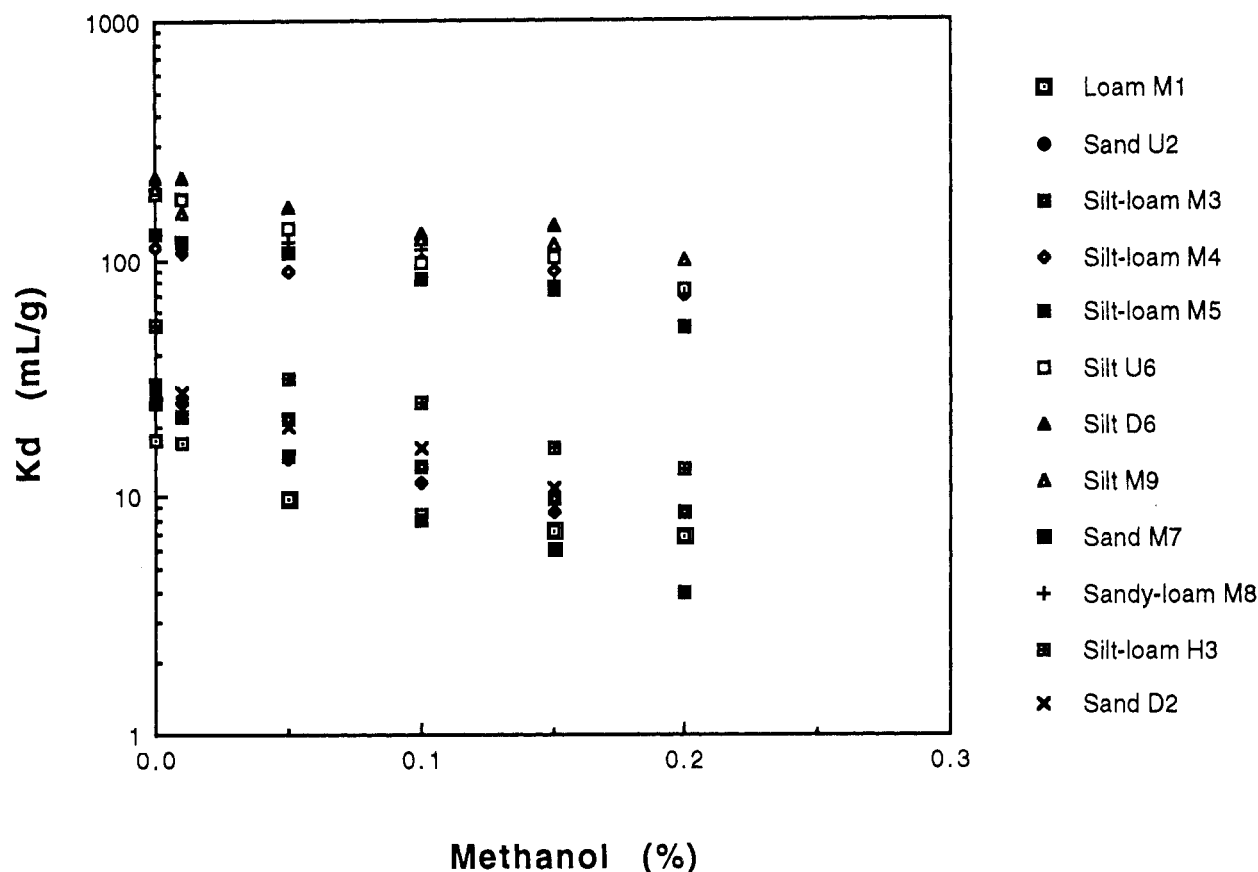
**Batch Sorption Studies.** From the log-linear relation between  $K_d$  and the fraction of cosolvent (Figure 2), distribution coefficients for water (no cosolvent) were generated (Table II). The  $K_d$  values for chlordane (18–220 mL/g) are indicative of its low solubility and strong affinity for the solid phase. Sorption of chlordane to the solid phase within the surficial aquifer beneath the dump may greatly restrict the mobility of this compound in groundwater at the site. The linearity of the sorption isotherms using a sand (sample 2;  $K_d = 23$  mL/g) and a silt (sample 6;  $K_d = 155$  mL/g) (Figure 3) sample are typical of the nonspecific solute–solid interactions of hydrophobic compounds (27, 28). However, it should be noted that such isotherms were obtained from a 5% methanol solution, which would enhance chlordane solubility in the aqueous phase, thus reducing apparent  $K_d$  values (Table II).

A plot of  $K_d$  versus the organic carbon fraction of the solid phase (Figure 4) resulted in a correlation of  $r^2 = 0.822$ . Such a relation was used to normalize distribution coefficients with respect to the organic carbon content of the solid phase:

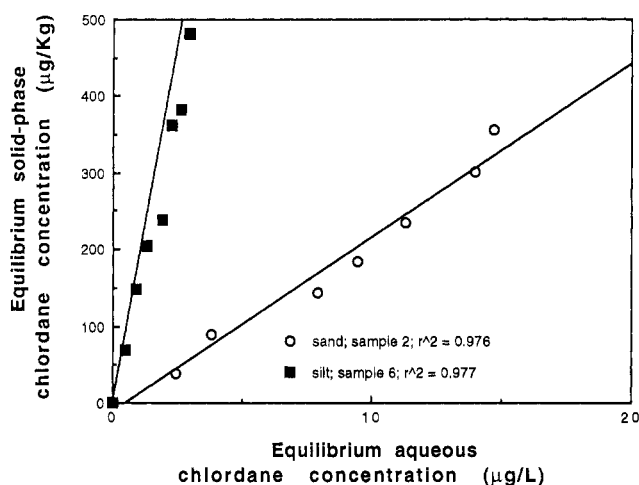
$$K_{\text{oc}} = K_d / f_{\text{oc}} \quad (2)$$

where  $K_{\text{oc}}$  is the organic carbon normalized partition coefficient and  $f_{\text{oc}}$  is the fraction of organic carbon in the solid material (Table II). Therefore, for a hydrophobic compound such as chlordane,  $K_{\text{oc}}$  values may be used to predict sorption on different solids of known organic carbon content. In this study, no correlation was found between  $K_d$  and particle size distribution of the solid phase. There is, however, some debate in the literature over the influence of mineral adsorption on the partitioning of nonionic compounds (29, 30), especially when the organic carbon content of the sample is low (typically <0.1%) (31, 32). The nature of the organic phase may account for the lack of correlation between solid-phase organic carbon content and  $K_d$  in some instances (33, 34). Chiou et al. (35) showed that the soil humic fraction was the major factor governing partitioning of nonionic compounds. The influence of the soil mineral phase was affected by the soil moisture and the nature of the aqueous phase.

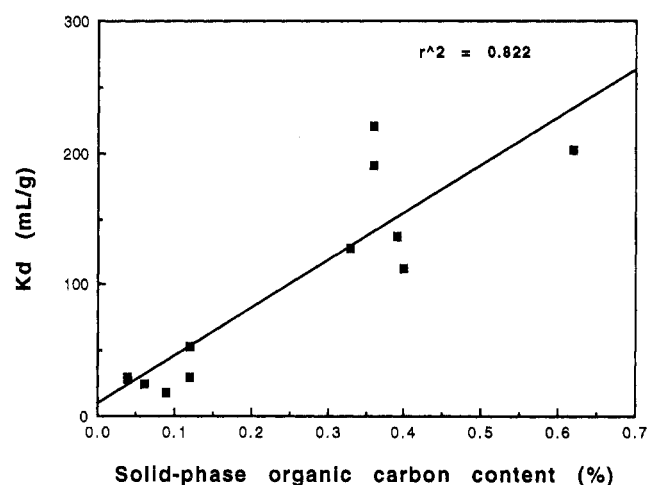
Although useful information could be obtained on the partitioning behavior of chlordane using the batch sorption technique, it is suspected that the method results in artificially low  $K_d$  values as a result of either particle interaction induced desorption processes (27, 36) or the hydrophobic compound remaining associated with a col-



**Figure 2.** Log-linear relationship between distribution coefficient,  $K_d$ , and the fraction of methanol in the aqueous phase for selected geologic materials from the North Hollywood Dump. Sample numbers are explained in Table II.



**Figure 3.** Sorption isotherms for chlordane with sand (sample 2) and silt (sample 6) from the North Hollywood Dump in a 5% methanol solution.



**Figure 4.** Relationship between distribution coefficient,  $K_d$ , and the fraction of organic carbon in geologic materials collected from North Hollywood Dump. Sample numbers explained in Table II.

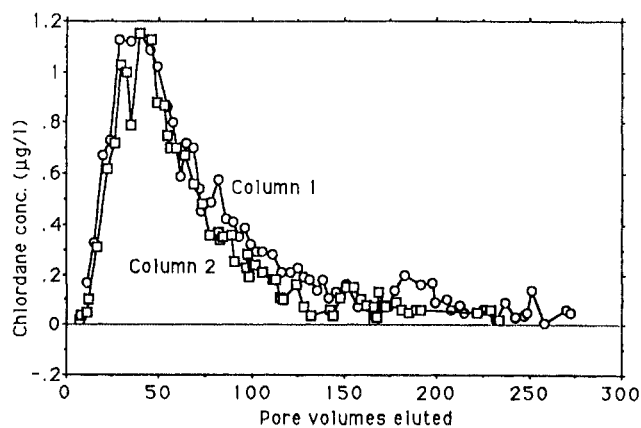
loidal phase (37, 38) following centrifugation.

**Column Studies.** Although residual methanol from the initial loading of chlordane to the column may have affected chlordane mobility, the column studies demonstrated the effect of humic substances on chlordane transport through a sand sample (Table II, solid 7) collected from North Hollywood Dump. When eluted with a commercial humic solution of 25 mg/L as DOC, the arrival of the chlordane breakthrough curve for duplicate columns occurred at approximately 20 pore volumes (Figure 5). When eluted with a commercial humic solution of 2.5 mg/L as DOC, the arrival of the chlordane breakthrough curve occurred at approximately 82 pore volumes (Figure 6).

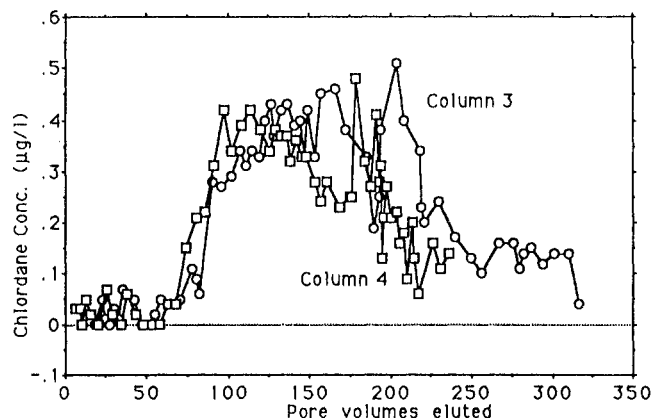
Due to chromatographic partitioning, the average velocity of a compound,  $V_c$ , can be retarded relative to the average velocity of water,  $V_w$ . In saturated groundwater systems, retardation of a compound is given by the equation (39)

$$R = V_w/V_c = 1 + K_d[\rho_s(1 - n)/n] \quad (3)$$

where  $R$  is the retardation factor,  $\rho_s$  is the density of the solid phase,  $K_d$  is the distribution coefficient for chlordane, and  $n$  is the porosity. To explain observations that compounds of low aqueous solubility are often more mobile than would be expected from simple chromatographic partitioning and data obtained in sorption experiments, Hutchins et al. (40) developed a modified form of the



**Figure 5.** Breakthrough curves for chlordane through columns containing sample 7 from North Hollywood Dump and eluted with humic substances of 25 mg/L as DOC.



**Figure 6.** Breakthrough curves for chlordane through columns containing sample 7 from North Hollywood Dump and eluted with humic substances of 2.5 mg/L as DOC.

**Table III.** Column Characteristics and Retardation Factors

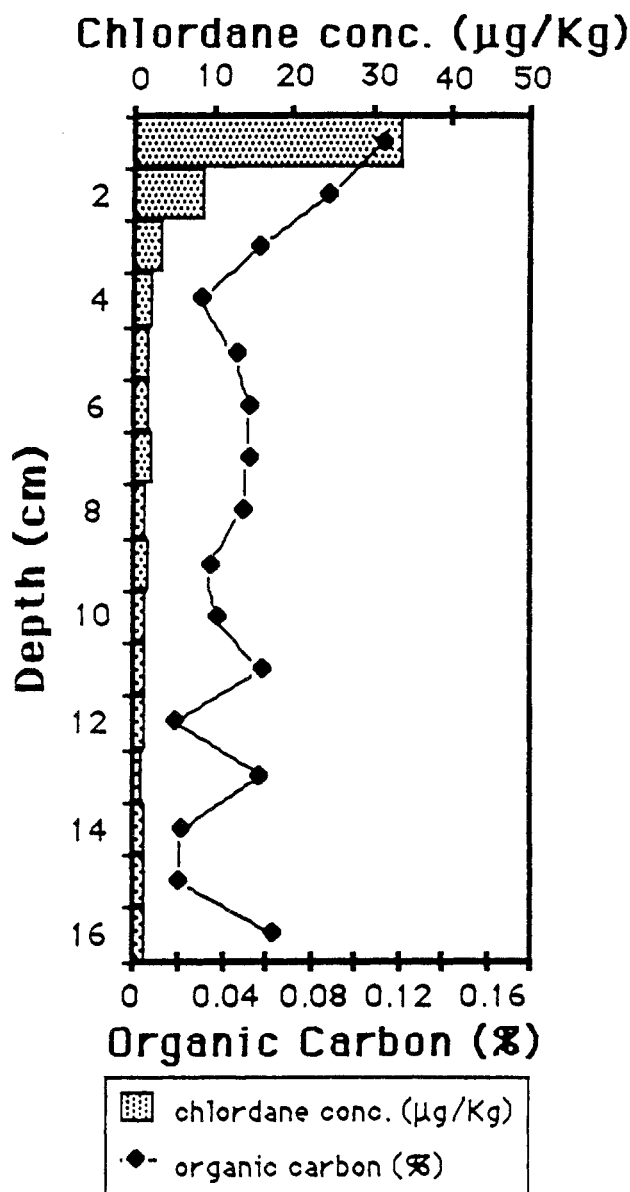
column	$K_d$ (mL/g)	$\rho_s$ (g/cm <sup>3</sup> )	$ n $	$K_{doc}$	DOC concn (mg/L)	retardation factors	
						obsd	pred
1	25	2.65	0.40	$10^{5.45}$	25	50	13
2	25	2.65	0.45	$10^{5.45}$	25	50	11
3	25	2.65	0.36	$10^{5.45}$	2.5	165	71
4	25	2.65	0.41	$10^{5.45}$	2.5	150	58

retardation equation that takes into account the effect of DOC:

$$R = 1 + \frac{K_d[\rho_s(1-n)/n]}{1 + K_{doc}x} \quad (4)$$

where  $K_{doc}$  is the partition coefficient based on DOC and  $x$  is the DOC concentration of the aqueous phase. By use of eq 4 and the values in Table III, predicted retardation factors for the columns eluted with humic solutions of  $25 \times 10^{-6}$  g/mL as DOC were 13 and 11 pore volumes. Predicted retardation factors for two columns eluted with humic solutions of  $2.5 \times 10^{-6}$  g/mL as DOC were 71 and 58 pore volumes. Observed retardation was somewhat greater than these predicted values (Table III).

The solubility experiments demonstrated a significant association of chlordane with organic matter in the aqueous phase. In the column studies, partitioning is taking place between three phases: water, dissolved humic substances, and the solid phase. Thus, if the mobile phase is augmented with humic substances, it is expected that chlordane mobility will be enhanced. In column studies with



**Figure 7.** Percent organic carbon content and chlordane concentration of the solid phase following column elution.

Lincoln fine sand, Kan and Tomson (41) showed that the retardation of phenanthrene was reduced from 90 to 4 pore volumes in the presence of a surfactant, 0.2% Triton X-100, as a model for dissolved humic substances.

The greater retardation of chlordane observed in this study may be caused by the retention of humic substances that form an organic coating on particles in the column. Indeed, for columns eluted with a humic solution of 25 mg/L as DOC, there was an increase in solid-phase organic content in the uppermost 2 cm of the column (Figure 7). Enfield et al. (24) demonstrated the facilitated transport of the hydrophobic compounds pyrene and hexachlorobenzene in the presence of organic macromolecules, and they concluded that the impact of humic acid on such transport was reduced, because it was itself retarded on passage through the soil columns.

### Conclusions

Batch solubility experiments showed an apparent increase in chlordane solubility with increasing concentration of humic substances. While solubility in pure water was 32 µg/L, solubility was enhanced to over 400 µg/L in the presence of extracted humic substances and to 146 µg/L

in groundwater collected downgradient of North Hollywood Dump. The distribution coefficients obtained for chlordane (18–220 mL/g) in batch sorption experiments illustrate the strong binding of chlordane to the solid phase. Column studies demonstrated that the mobility of chlordane is enhanced when the aqueous phase contains humic substances. Chlordane mobility was somewhat less than that predicted by a modified form of the retardation equation. This decreased mobility may be due to the retardation of humic acid within the column. The effect of humic substances on the partitioning and mobility of chlordane is important because groundwater downgradient from the North Hollywood Dump has concentrations of aqueous organic carbon that can significantly increase chlordane concentration in the mobile phase.

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**Registry No.** Chlordane, 12789-03-6; carbon, 7440-44-0.

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