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Effect of Sewage Sludge on Trace Element Mobility in Soils

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ABSTRACT

Adsorption of Be, F, B, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Se, Sr, Mo, Tc, Ag, Cd, Sn, Sb, Cs, Ba, Hg, Pb, Bi, and P was measured in a sandy soil and a sandy loam soil at concentration levels closely corresponding to those to be expected for field conditions. Mobilities of the elements in the soils were calculated from the adsorption data. Of the trace elements studied, F, B, and Tc were found to be very mobile in both the sandy and sandy loam soils. Manganese, Sr, and Sb were highly mobile only in the sandy soil, and Mo in the sandy loam soil. In general, sludge solutions appeared to increase the mobility of elements in a soil. This is due to a combination of complexation by dissolved organic compounds, high background concentrations, and high ionic strengths of the soil solutions. The relative effects of these factors vary strongly among elements.

Equations were derived predicting the rates of accumulation in soils and accompanying increases in the soil solutions of trace elements added with sewage sludge.

When adsorption was related to soil organic matter content, for many trace elements the strength of adsorption was found to depend only on pH, increasing with increasing pH.

Additional Index Words: **heavy metals, adsorption, organics, soil.**

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Land application is a method frequently used to dispose of sewage sludge. The beneficial effects of sewage sludge on soil fertility will, however, be more than offset by accumulation of toxic elements in the soil if land disposal is not carefully controlled (De Haan, 1978; Chang et al., 1981). Perhaps in the near future guidelines for the disposal of sewage sludge will be set in such a way that the maximum permissible loading of a soil with sludge will be determined by the original level of trace elements in the soil (Purves, 1979). This level will then be allowed to increase by a certain factor depending on the adsorption characteristics of the soil. Present practices of land application of sewage sludge (De Haan, 1978; Chang et al., 1981) are leading to a steady accumulation of a number of trace elements in the soil.

Although trace elements are assumed to be retained in soils, leaching of a number of trace elements in sludge-amended soils has been shown (Poelstra and Frissel, 1979; De Haan, 1980). To estimate the movement of heavy metals and other toxic elements associated with sewage sludge in soils, data on physical and chemical parameters concerning the soil adsorption processes are essential. In addition, although not discussed in this paper, factors such as erosion, tillage, uptake and removal with crops, and possibly biomethylation (Brinckman and Bellama, 1978) must be considered in a complete description of the mobility of elements in soils. Adsorption is affected by speciation of the elements in the soil solution and by pH, E_h, ionic strength, and composition of the soil solution, as well as by the clay and organic matter content of the soil (Wolf et al., 1977; McLaren et al., 1981). Many adsorption studies in soils pertain to concentration levels much higher than those to be expected under field conditions (Jarvis and Jones, 1980; Jarvis, 1981). Adsorption data for conditions in soils are indispensable for the proper modeling of the mobility of trace elements in soils (Christensen, 1981).

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Table 1—Cationic composition of aqueous solutions used in adsorption experiments with soils.

Composition	Solution phase†								
	10	11	12	20	21	22	30	31	32
	meq/L								
NH_4^+	0	0	0	60	11	0.23	65	7	0.12
Ca^{2+}	0	3	30	5	16	5	5.2	30	12.8
Mg^{2+}	0	0	0	0.6	3.2	0.8	0.7	6	1.4
Na^+	0	1	10	4.8	5.2	0.6	8	7.3	1.1
K^+	0	1	10	2.1	2.3	0.3	2	2	0.44
Total cations	0	5	50	72.5	37.7	6.9	87.8	52.3	15.9
pH	‡	--	--	7.7§	6.4	5.2	7.6§	5.8	5.6
$\frac{Ca+Mg}{Na+K+NH_4}$	--	1.5	1.5	0.08	1.04	5.1	0.08	2.2	8.5

† Solution phase: 10 = deionized water; 11 and 12 = salt solutions; 20 = supernatant of industrial anaerobically digested sludge; 21 = idem after aeration of the sludge; 22 = idem of sludge solids which, after aeration and centrifugation were resuspended in water and aerated; 30, 31 and 32 = the same as for 20, 21, and 22, but obtained from domestic sludge.

‡ Not detectable.

§ Before equilibration with the sandy soil, the pH was adjusted to between 5 and 6 with HNO_3 to eliminate carbonate.

Our paper gives information on the physicochemical distribution (adsorption) of 26 elements, added as inorganic species, between the solid and liquid phases in two types of soils and in sewage sludges.

METHODS AND MATERIALS

Eighteen soil/soil-solution phase systems resulting from the combination of two soils with nine liquid phases were used to determine the distribution between soil and soil solution of Be, F, B, V, Cr, Mn, Fe, Co, Ni, Zn, Cu, As, Se, Sr, Mo, Tc, Ag, Cd, Sn, Sb, Cs, Ba, Hg, Pb, Bi, and P. The liquid phases used for equilibration of the soils were distilled water (one time), salt solutions of different ionic strengths (two times), and solution phases of sewage sludges (six times). The ionic strength of the salt solutions was adjusted at two levels: 0.0035*M* or 0.035*M* ($CaCl_2$ + NaCl + KCl) (3:2:2). Sludge solution phases were obtained after centrifugation at 40.000 *g* for 1 hour (MSE 18 high-speed, 17,000 rpm; MSE Scientific Instruments, England.). Industrial and domestic anaerobically digested sludges were used. Data on the elemental composition of these sludges, typed "Almelo" and "Alkmaar," have been published (De Haan, 1978). For a number of elements (Be, V, Se, Sr, Tc, Ag, Sn, Sb, Cs, and Bi), however, data on total concentrations had to be estimated from the literature (Furr et al., 1976). Ten liters of the sludges were aerated intensively for several months in glass fermentation vessels, with constant mechanical stirring. The total volumes of the sludges were kept constant by adding water. After centrifuging and decanting the solution phase, the remaining sludge solids were made up with water to the original volume of 10 liters.

After an equilibration period of between 2 and 4 weeks, with constant stirring and aeration, sludge supernatants were again collected after centrifugation. The compositions of the solution phases are given in Tables 1 and 2. Dry matter contents of the sludges, determined after drying at 105°C for 20 hours, ranged from 1 to 3%. Organic matter, determined as weight loss after ashing at 600°C for 4 hours, corrected for carbonate, was 50-60% of total dry matter.

Basic characteristics of the two soils used, a sandy soil and a sandy loam soil, are given in Table 3. The soils differ mainly in pH and clay contents. Organic matter contents and cation exchange capacities (CEC) are similar. The soils were stored in polythene bags at 2°C. All labware used was made of polythene or polypropylene and was soaked in 10% HNO_3 and rinsed with doubly distilled water before use.

Soil adsorption was determined with a series of solutions where the elemental concentration ranged from 0 to 5 ppm above the concentration of the initial solution. A radioactive tracer of the element studied and 5 g soil were added to 25 ml of these solutions. The soil/solution mixtures were equilibrated for 3 days at 20°C on an orbital shaker. Soil solutions were obtained after centrifugation at 40,000 *g* for 1 hour. The radioactivity of the solution phase was measured in 20-ml vials in a liquid scintillation counter (Philips) for ^{63}Ni and ^{99}Tc and a gamma counter (Philips) for all other radioisotopes used. The distribution constant (K_d), defined as the ratio between the increase in the amount of element adsorbed per unit weight of soil and the accompanying increase in soil solution concentration, can be calculated from the measured data according to:

Table 2—Anionic/organic composition of aqueous solutions used in adsorption experiments with soils.

Composition	Solution phase†					
	20	21	22	30	31	32
	mg/L					
Organic N	50	30	7	50	10	1
NO_3-N	--‡	30	40	--	500	80
NO_2-N	--	5	--	--	1	--
Inorganic P	70	15	20	25	100	40
Organic P	n.s.‡	n.s.	n.s.	n.s.	n.s.	n.s.
Cl	200	200	10	250	250	25
Organic C	250	400	n.d.‡	250	400	n.d.
Sulfide S	0.1	--	--	0.1	--	--
SO_4-S	--	30	n.d.	--	50	n.d.

† Solution phase: 20 = supernatant of industrial anaerobically digested sludge; 21 = idem after aeration of the sludge; 22 = idem of sludge solids which, after aeration and centrifugation were resuspended in water and aerated; 30, 31, and 32 = the same as for 20, 21, and 22, but obtained from domestic sludge.

‡ n.s. = not significant; n.d. = no determination; -- = not detectable.

$$K_d = \Delta C_s/\Delta C_w = (C^*_{tw} - C^*_w)V_w/(C^*_w G_s) \quad \text{(ml/g)}, \qquad [1]$$

in which:

C^*_{tw} = concentration of added radioisotope in the solution phase if no adsorption occurred, measured in a blank without soil (cpm/ml);
C^*_w = concentration of the radioisotope in the solution phase after 3 days equilibration with soil (cpm/ml);
V_w = volume of solution phase phase (ml);
G_s = mass of the solid phase (g);
ΔC_s = increase in concentration of the element in the soil solid phase (μg/g); and
ΔC_w = corresponding increase in concentration of the element in the solution phase (μg/ml).

The specific radioactivity and amounts of radioisotopes added were such that no significant increases in the background concentrations of the elements in the various soil solutions resulted. The distribution constant (K_d) could thus be determined at the natural background concentrations of the elements ($\Delta C_w \sim 0$). Assuming K_d to be constant (K_o) in this region, its relationship with the migration velocity of the element in the soil can then be determined from:

$$V_X = \frac{V_{H_2O}}{(l + \omega)K_o} = \frac{R}{\Phi + \delta K_o}, \qquad [2]$$

in which:

V_X = migration velocity of an element with a distribution constant K_o in the soil (mm/year),
V_{H_2O} = linear velocity of water in the soil (mm/year),
R = surplus annual rainfall (mm/year),
Φ = soil moisture content (cm^3/cm^3),
δ = soil bulk density (g/cm^3), and
$\omega = \delta/\Phi$ = phase ratio in a volume of soil (g/cm^3).

Table 3—Characteristics of soils used in the adsorption experiments.

Soil type	pH (H_2O)	Moisture (of oven-dry wt)	Organic matter†	Clay†	CEC
		g/g			meq/g
Sandy	5	0.11	0.035	0	0.22
Sandy loam	8	0.25	0.025	0.2	0.16

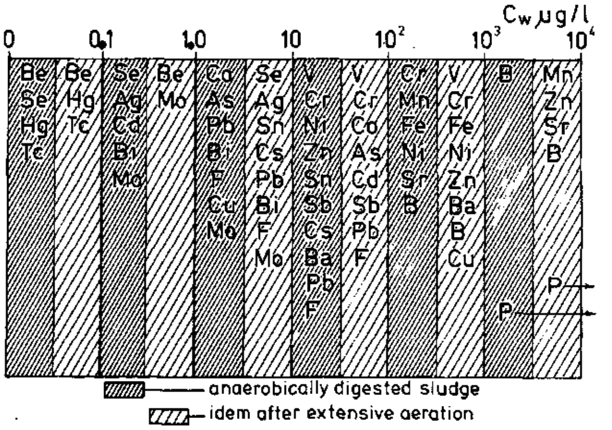

Fig. 1—Solution concentration ranges of elements in sewage sludge.

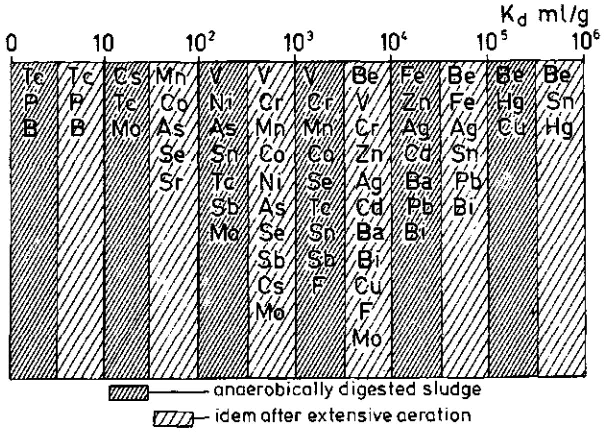

Fig. 2—Distribution constant ranges of elements in sewage sludge.

The radioactive isotopes were obtained as chlorides or nitrates (the Radiochemical Centre, Amersham) in the valence states most likely to occur in aqueous solutions (pH = 4–8) under atmospheric pressure and O_2 level: $^{7}Be^{2+}$, $^{48}VO^{2+}$, $^{51}Cr^{3+}$, $^{54}Mn^{2+}$, $^{59}Fe^{3+}$, $^{58}Co^{2+}$, $^{63}Ni^{2+}$, $^{65}Zn^{2+}$, $^{74}AsO_4^{2-}$, $^{75}SeO_3^{2-}$, $^{85}Sr^{2+}$, $^{99}TcO_4^{-}$, $^{110m}Ag^{+}$, $^{109}Cd^{2+}$, $^{113}Sn^{4+}$, $^{125}Sb^{3+}$, $^{137}Cs^{+}$, $^{133}Ba^{2+}$, $^{210}Pb^{2+}$, and $^{207}Bi^{3+}$.

The distribution behavior of Cu, Hg, Mo, B, F, and P was studied without radioactive tracers. These elements, added in standard solutions (Merck Titrisol) as Cu^{2+}, Hg^{2+}, Mo^{6+}, B^{3+}, F^-, and PO_4^{3-}, were determined directly in the sludge solution phase. Mercury was determined after reduction by atomic absorption spectrophotometry in a gas flow-through cell; Cu was preconcentrated by extraction and determined similarly using a graphite furnace. Molybdenum, boron, and phosphorus were determined colorimetrically. Fluoride was determined potentiometrically with a selective membrane electrode. In addition, solution concentrations of Pb, Zn, and Cd were also determined directly by anodic stripping voltammetry (PAR analyzer 174 A) in a background buffer at 0.05*M* Na-acetate (pH = 4.5). Blanks were run throughout all analyses. The concentration ranges of all other elements were calculated indirectly from the distribution constants (Fig. 2) and the total concentrations of the elements in the sludges.

Without radioactive tracers K_d follows directly from:

$$K_d = C_s/C_w = (\Delta C_{tw} - C_w)V_w/(C_w G_s) \quad (\text{ml/g}), \qquad [3]$$

in which ΔC_{tw} = the concentration of the element added to the solution phase if no adsorption occurred, measured in a blank without soil (µg/ml).

Working without radioisotopes usually entails a significant increase of the concentration of an element in solution before a measurement can be made. The distribution constant at the background concentration of the element in the soil solution was found by plotting ΔC_s against ΔC_w and extrapolating K_d to $\Delta C_w = 0$. In the same way distribution constants of the elements (K_o values) were also measured in the anaerobically digested and aerated sewage sludges. After addition of the element or isotopic tracer element, the sludges were left to stand for 1 month before analyses were made. The aerated sludges were oxygenated once a week (E_h >200 mV).

RESULTS AND DISCUSSION

Ranges of elemental concentrations in the various sewage-sludge solution phases are given in Fig. 1; ranges of distribution constants are given in Fig. 2. As can be seen, aeration with accompanying changes in pH and ionic composition of the solution phase (Tables 1 and 2) greatly influences the distribution and, in general, increases the solution concentrations of elements in sludges. The E_h of the aerated sludges was ≥200 mV, and of the anaerobic sludges < −300 mV. After sampling the sludge for distribution experiments the E_h was found to rise to between −300 and 0 mV. This no doubt accounts for the behaviour of Tc in the anaerobically digested sludges (Fig. 2), in which K_d was found to vary between 10 and 10,000. Reduced forms of Tc are strongly adsorbed to organic matter (Balogh and Grigal, 1980; Mousny and Myttenaere, 1981) and their rates of formation can vary greatly in the observed E_h range of 0 to −300 mV.

Except for P, the concentrations of the elements determined directly in the sludge solutions were found to be within the ranges calculated indirectly from the distribution constants and total elemental concentrations in the sludge. This shows that distribution of trace elements in sludges is a rapid process involving all species of an element and ruling out slow precipitation reactions. In the case of P, calculated concentrations were much higher than actual concentrations. Phosphorus is a major element in soils and sludges and is known to be involved in slow precipitation reactions (Beek and Van Riemsdijk, 1979). The distribution constants measured after 3 days will thus be nonequilibrium values, explaining the observed discrepancy.

If adsorption is rapid and reversible K_o governs the basic mobility of trace elements as expressed by Eq. [2] (Huber and Gerritse, 1971, 1973). When several species of a trace element are present, Eq. [2] also applies as long as the species are in mutual rapid equilibrium. If not, each species must be treated separately and kinetic parameters must be determined for proper calculation of mobility. If adsorption itself is slow, as in the case of P, Eq. [2] can be used in conjunction with a kinetic model to predict elemental mobility (Gerritse et al., 1981; Gerritse, 1981).

Examples of isotherms are shown in Fig. 3 for Cd, Se, and Cu. Rather than presenting all individual isotherms (468 in all), the ranges of distribution constants (K_o's) for each element found with the water and salt solutions (white bars) and the ranges found with the six sludge solutions (dark bars) are given in Fig. 4 and 5. The ranges of relative elemental mobilities ($V_{rel} = V_X/V_{H_2O} \times 100\%$) are also shown in Fig. 4 and 5 and were calculated with Eq. [2]. In most cases the sludge solutions appear to increase the mobility of the elements. A striking example is Sn (Fig. 4 and 5). This is due to a combination of complexation by dissolved organic compounds, high background concentration (e.g., in the cases of B, Mn, Zn, Sr, and P) and high ionic strength

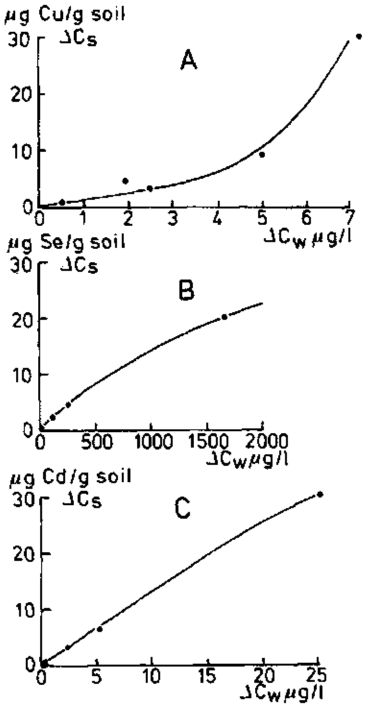

Fig. 3—Examples of adsorption isotherms found for the sandy loam soil using 0.0035*M* ($CaCl_2$ + NaCl + KCl) (3:2:2) as equilibrating solution.

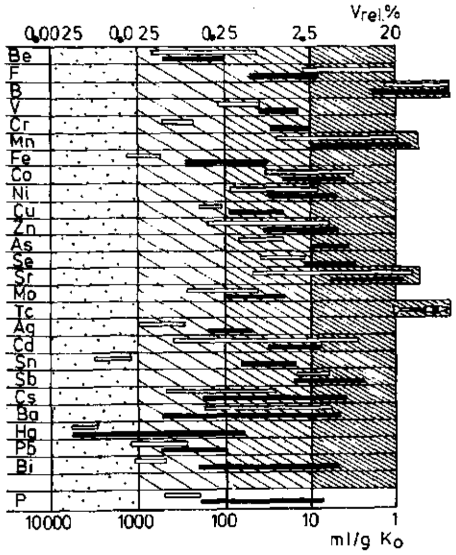

Fig. 4—Trace element mobility in a sandy top soil. White bars represent V_{rel} or K_o found after equilibrating the soil with inorganic solution phases. Dark bars represent the range found after equilibrating with sewage sludge solution phases.

of the equilibrating solution. The relative effects of these factors will vary strongly among elements. Highly mobile elements (possible velocity relative to water in the soil is > 10%) are F, B, Mn, Sr, and Sb in the sandy soil and F, B, and Mo in the sandy loam soil. Technetium was only slightly adsorbed in the sandy soil and barely detectable in the sandy loam soil, indicating its high mobility. Under anaerobic conditions in the soils, however, the mobility of Tc is drastically reduced (Balogh and Grigal, 1980; Mousny and Myttenaere, 1981). Mobility of most elements in the sandy loam soil is lower than in the sandy soil. Molybdenum, phosphorus and technetium appear to be the only exceptions. The relationship between K_o and the average annual distance migrated into the soil by an element is given by Eq. [2].

Due to dispersion the distance migrated by an element in the soil will be spread around an average distance λ. For a linear isotherm this scatter will be proportional to the dispersion length (DISP) via $(\lambda \times \text{DISP})^{1/2}$ if DISP $\ll \lambda$ (Bolt, 1979). Dispersion length is a soil characteristic and usually about 1-5 cm when measured in soil columns. It can, however, be as much as 20-40 cm under field conditions (Van Hoorn, 1981). If DISP ~ λ and/or the distribution isotherm is nonlinear, the contribution of dispersion can be estimated graphically (Bolt, 1979) or by computer simulation (Christensen, 1981; Gerritse et al., 1981).

The accumulation of trace elements in the tillage layer of a soil through sludge disposal can, for a linear distribution isotherm, be related to the distribution constants given in Fig. 4 and 5 with the equation:

$$\Delta = 100\,(\varrho X/R)\,[(1 + \omega K_o)/\omega]\,q\,(1 - q^n), \quad [4]$$

for $0 \leq q < 1$ and $\Delta = 0$ for $q \leq 0$, and in which:

Δ = increase in total trace element concentration in the tillage layer of the soil (µg/kg) after n years,
ϱ = trace element concentration in the sludge (mg/kg of oven-dry matter),
X = annual application of sludge (t dry matter/ha),
R = surplus annual rainfall (mm),
n = number of years of addition, and
$q = 1 - (10R/y)[\omega/(1 + \omega K_o)]$, y being mass (t) of 1 ha of plowed soil (tillage layer).

The accompanying concentration of a trace element in the soil solution is given by:

$$C_w = \Delta\,[\omega/(1 + \omega K_o) = 100(\varrho\,X/R)\,q\,(1 - q^n). \quad [5]$$

Equations [4] and [5] are derived with simple mathematics from the distribution constant as used in Eq. [1-3].

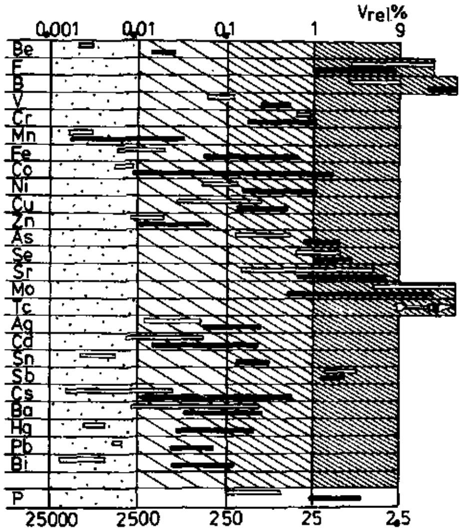

Fig. 5—Trace element mobility in a sandy loam top soil. As for Fig. 4.

Table 4—Distribution constants and ranges calculated for two highly organic soils, one unpolluted (*A*) and the other polluted with heavy metals (*B*);† a sandy soil (*C*) and a sandy loam soil (*D*);‡ two peats;§ and anaerobically digested sewage sludges‡ before (*1*) and after (*2*) aeration.

	K_d^*							
	Soil A	Peat A¶	Soil C¶	Sludge 2	Peat B#	Soil B	Soil D¶	Sludge 1
pH	4.5	4 - 5	4.5 - 5.0	5 - 6	6	6.2	7.5- 8	7- 7.7
Organic matter, %	90	>90	3.5	60	>90	>90	2.5	60
	L/g organic matter							
V	5.6	--	1 - 4	1 - 2	--	1.3	8 - 15	1- 2
Cr	6.7	--	6 -20	1 - 10	--	41	1 - 1.5	2- 4
Mn	1.1	--	0.03- 1	0.05- 0.2	--	15	400 -600	2- 5
Ni	1.1	--	0.3 - 3	0.2 - 1	--	5.2	9 - 18	1
Cu	4.9	1 -50	3 - 6	5 - 10	19	37	5 - 50	150-250
Zn	2.1	0.07-15	0.2 - 4	3 - 4	--	3.2	70 -100	60- 90
Cd	1.6	0.02-20	0.07-13	2 - 3	--	6.4	20 -150	80-120
Pb	28	0.2 -70	8 -37	90 -120	--	26	120 -160	100-150

† From data of Tyler (1978).
‡ This paper.
§ *A*: Wolf et al. (1977); *B*: McLaren et al. (1981).
¶ Distribution constant ranges calculated for inorganic phase solutions only ([Ca^{2+}] = 0-0.015*M*).
# Measured in 0.05*M* $CaCl_2$.

As can be expected, the rate of accumulation of an element as well as the maximum level reached in the soil is dictated by the amount added annually and the distribution constant.

To illustrate the importance of organic matter, data from the literature (Wolf et al., 1977; Tyler, 1978; McLaren et al., 1981) are compared with the distribution constant ranges found for the soils and sewage sludges described in this paper (Table 4). The distribution constants are given per unit weight of organic matter instead of oven-dry weight of soil or sludge. The relationship between these distribution constants is given by:

$$K_d = K_d^*(A/100), \qquad [6]$$

in which:

K_d = distribution constant (ml/g) per unit weight of soil or sludge dry matter,
K_d^* = distribution constant per unit weight of soil or sludge *organic matter,* and
A = organic matter in percent of soil or sludge dry matter.

In this way it can be seen that for many of the elements considered in Table 4 the order of magnitude of the distribution constants is more or less independent of the type of phase system, but dependent on pH. Especially in the pH range of 4 to 6, agreement is good. The alkaline soils and sludges show much less agreement. This is due to the much greater competition in this pH region between adsorption to solid organic matter and complexation by dissolved organic matter. Anionic species, however, (e.g., the predominant forms of B, Tc, As, Se, Mo, fluoride, and cations such as Cs^+ that do not readily form complexes) show much less affinity for soil organic matter.

Sludge organic matter can add to the adsorption capacity of a soil for trace elements. A large part (50-80%) of organic matter of stabilized sewage sludge is fairly resistant to decomposition in the soil (Terry et al., 1979a, 1979b) and is similar to humified organic matter. However, when the sludge is added to soil, part of the native soil organic matter is decomposed (priming effect; Terry et al., 1979a, 1979b). With recommended sludge disposal rates in The Netherlands (1-2 t dry matter/ha per year; De Haan, 1978), the effect of added sludge on the soil adsorption capacity may be assumed to be small, and, based on organic matter (Van Dijk, 1980), the extent of adsorption will hardly change for many trace elements (Table 4, Eq. [6]).

The problem of accumulation accompanied by increasing levels of trace elements in the soil solution, however, remains and will increasingly present itself in the future, as can be concluded from the data and literature given in this paper.

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