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For Reference

May 1990

DISCLAIMER

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SOIL SELENIUM FRACTIONATION, DEPTH PROFILES AND TIME TRENDS IN A VEGETATED UPLAND AT KESTERSON RESERVOIR

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ABSTRACT. Kesterson Reservoir, in the San Joaquin Valley of California, was used for disposal of saline, seleniferous agricultural drain waters during the years 1981 - 1986. The combined effects of infrequent drain water application, reduction and oxidation of Se, and evapotranspirative removal of soil water within the upper 1 m of the soil profiles appear to provide explanations for distribution patterns of Se. From 1987 to 1990, an approximate doubling in average soil solution Se concentrations was observed. It is shown that currently only relatively insignificant quantities of Se were transported into these Pond 11 soils from the shallow water table. Sequential extraction of these upland soils indicated that substantial inventories of Se were associated with *soluble, adsorbed, carbonate,* and *soil organic matter* fractions, while Se retained within refractory inorganic fractions were minor. The mobilization of Se from these potentially labile pools may account for the observed increases in soil solution concentrations of Se following the draining of Kesterson Reservoir.

1. INTRODUCTION

Kesterson Reservoir, once a set of evaporation ponds for agricultural drain waters, is situated in the southern portion of the Kesterson National Wildlife Refuge, on the west side of the San Joaquin Valley, California. The Reservoir was comprised of 12 ponds, totaling 520 ha. Seleniferous drain waters were delivered to Kesterson via the San Luis Drain, from farmlands approximately 140 km further south in the western San Joaquin Valley. Approximately 9 Mg of Se, and 3×10^5 Mg of salts were delivered to Kesterson between 1981 and 1986 (U.S.B.R., 1986). Drain water and pond water Se occurred primarily as selenate (Se(VI)) under the prevailing oxidizing, neutral to slightly alkaline surface water environment. The salt composition of these waters was dominated by Na^+, SO_4^{2-}, and Cl^-. While approximately 50% of the salt load was diminished through seepage losses, Se inputs were largely confined to the surface and near surface environment (Weres et al., 1989). Reducing conditions which developed during drain water ponding removed Se from solution through transforming the highly soluble Se(VI) pool to a combination of primarily less soluble species including selenite (Se(IV)), elemental Se, and various organic associations (Weres et al., 1989).

Studies at Kesterson conducted by the U.S. Fish and Wildlife Service from 1982 through 1985 indicated that high concentrations of Se brought in by the drain waters were responsible for waterfowl deformities and nesting failures (Ohlendorf, 1989). In response, drain water disposal at Kesterson was terminated in 1986. Presently, the Reservoir is being maintained as an upland environment, with continuing monitoring of groundwater, soil, vegetation, and wildlife, and continued research on remediation through microbial volatilization of Se (Frankenberger and Karlson, 1989; Weres et al.,1989).

A large inventory of selenium remains in Kesterson soils where approximately 75% is concentrated in the upper 0.15 m of soils. Se concentrations in the surface soils ranged from 1 to over 100 mg kg^{-1}, with a Reservoir-wide average concentration of 5 mg kg^{-1}. Only 5 to 20% of Se in the detrital layer and surface soils is readily water-soluble. Nevertheless, extremely high concentrations of dissolved Se (1000 to 10,000 μg L^{-1}; the E.P.A. drinking water standard for Se is 10 μg L^{-1}.) are present in soil solutions under present conditions, down to depths of a meter below the soil surface. The objectives of the investigations described here include (1) monitoring temporal and spatial trends in soil Se concentrations and (2) determining the distribution of Se within various soil fractions. This paper focuses on distributions of Se in a *D. spicata* -dominated upland environment, one of the more stable and dominant upland environments at Kesterson. This data will provide information useful in assessing processes contributing to the future evolution of the Se inventory at Kesterson Reservoir.

2. METHODS

Characterization of Se in this upland habitat was initiated in January 1987 in a region designated as P11. These soils formed on old alluvial floodplains (of primarily Sierran granitic parent material, low in Se) of the San Joaquin River. The series was generally associated with poorly drained, saline, and occasionally sodic conditions. Low mean annual precipitation (290 mm), high potential evaporation (1800 mm y^{-1}), and the presence of a shallow water table commonly associated with saline soils are characteristic of the Reservoir. At the Pond 11 study area, the water table has fluctuated annually between 1.0 and 2.5 m below the soil surface during the interval spanning summer 1986 to spring 1990. These annual water table fluctuations have been correlated with winter flooding and spring draining of adjacent wetlands. It is worth noting that the soils of the Kesterson area were diagnosed as being saline long before development of the Reservoir and subsequent accelerated importation of salts (Cole et al., 1952). While the area soils have historically been saline, high concentrations of Se in these soils are solely due to the recent influx of drain waters.

2.1 Soil Profile Characterization, Monitoring, Soil and Soil Solution Sampling

Soil samples used for Se extraction studies were obtained by hand augering at 0.10 m intervals down to 1.70 m below the soil surface. Physical characterization of the soils included particle-size analyses, identification of evaporite and clay minerals, and measurements of hydraulic conductivities. The monitoring of soil solution profiles began in late January 1987, using tensiometers and soil solution samplers (0.15, 0.30, 0.46, 0.61, 0.91, and 1.22 m). Three similar test plots, designated P11C, P11D, and P11H, were instrumented in this manner. Additional test plots were constructed in which the surface 0.15 m and 0.30 m of soil were removed (designated P11S1/2 and P11S1 respectively). Previously installed shallow wells were used to monitor depths to the water table. In subsequent years, instrumentation at one plot was expanded to include additional soil solution samplers down to 1.50 m, and a set of 3 neutron probe access tubes (down to at least 1.50 m) for monitoring changes in soil water content. Soil solution collections were generally limited to the months of January through April, during which time the soil profiles were sufficiently saturated by rainfall and by the season rise of the shallow water table. Soil solution samples were routinely analyzed for Se(IV) and total solution Se by hydride generation atomic absorption spectrometry, HGAAS, (Weres et al., 1989).

The soils in the P11 test area were relatively homogeneous with respect to physio-chemical, characteristics when compared with other Reservoir sites. Clay and silt contents throughout the profile were in the range of 23±4% and 24±5% respectively (sandy loam to sandy clay loam U.S.D.A. textures). Field-saturated hydraulic conductivities ranged from 2×10^{-8} to 6×10^{-7} m s^{-1} in 15 replicated measurements within the upper 1.3 m of the site. The site is very saline, with all soil solutions and the shallow groundwater characterized by EC values exceeding 14 dS m^{-1}, sodium adsorption ratios (SAR) exceeding 30, and

slightly alkaline pH.Gypsum and calcite are distributed over the upper 1.2 m of the profile (Flexser, 1988). Roots of *Distichlis, Cressa,* and *Frankenia* permeate the upper 1.0 m of the soil. A distinct surface salt crust was absent at the P11 area, unlike the more frequently ponded areas of the Reservoir.

2.2 Sequential Extraction of Soil Selenium

Conventional sequential extraction techniques that have commonly been employed for trace metal fractionation in soils and sediments (Tessier, 1979; Chao, 1984) are inappropriate for elements like Se that can be released from a solid phase and readsorbed by undissolved surfaces during a particular extraction, and which can undergo oxidation state changes in others (Gruebel et al., 1989). As a result, a sequential extraction procedure developed by Lipton (1990) for specifically fractionating soil Se in arid, alkaline soils was used in this study.

Soils from site P11C were air-dried and ground to pass a 0.5 mm sieve for the sequential extraction. A soil:solution ratio of .1:10 (2.5 g/25 mL) was used for all extractions except for the NaOCl boil, used to remove soil organic matter (SOM), which employed a 1:4 ratio. Supernatant solutions were collected by centifugation (20,000 x g, 10 min) and the soil residue was rinsed with 0.25 mol L^{-1} KCl after each step. The rinses were combined with the preceeding extraction, filtered (0.45 μm), and analyzed for Se (by HGAAS) and other elements (by inductively coupled plasma spectrometry) within 2 weeks of collection.

The fractions of Se that were isolated are presented in the order extracted;

Soluble: 0.25 mol L^{-1} KCl (2 h) (Fujii et al., 1989). *(KCl)*

Adsorbed: 0.1 mol L^{-1} K_2HPO_4 (pH 8, 20 h). *(P8)*

Carbonate: 1 mol L^{-1} Na-acetate (pH 5, 1 + 4 h) followed by 0.1 mol L^{-1} K_2HPO_4 (pH 8, 20 h). *(OAc/P8)*

Soil Organic Matter *(SOM)*: NaOCl (pH 9.5, 0.5 h boil, repeated once) (Anderson, 1963). *(NaOCl)*

Easily Reducible Oxides *(ER-Ox)*: 0.1 mol L^{-1} NH_2OH (pH 2, 0.5 h) followed by 0.1 mol L^{-1} KOH (20 h).

Amorphous Oxides *(Am-Ox)*: 0.25 mol L^{-1} NH_2OH/HCl (0.5 h @50° C) followed by 0.1 mol L^{-1} KOH (20 h).

Crystalline Oxides *(Cryst-Ox)*: 4 mol L^{-1} HCl (0.5 h boil) (Chao and Sanzalone, 1989).

Amorphous Aluminosilicates *(Alk-sol Al/Si)*: 0.5 mol L^{-1} NaOH (0.2 h boil) conducted following *SOM, Am-Ox,* and *Cryst-Ox* extractions.

It must be emphasized that the names applied to the isolated Se fractions represent the best estimates of the pools of Se that would be removed by the respective extractant and do not imply perfect selectivity. For example, Se associated with the SOM (*SOM*-Se) may include various forms of Se that can be intimately associated with SOM; organic Se, elemental Se and adsorbed Se(IV) that may be protected from being released into the preceeding extractions by a "protective" coating of SOM. Organic Se removed by *P8* extractants was determined using the hydrophobic XAD-8 resin and subtracted from the total Se measured in the *adsorbed* and *carbonate* pools, and added to the *SOM*-Se pool as described by Lipton (1990). For a more detailed and critical discussion of the sequential extraction procedure and the associations of soil Se with the isolated fractions, the readers are referred to the above paper.

3. RESULTS AND DISCUSSION

3.1 Trends of soil solution selenium concentration

Depth profiles of soil solution Se concentrations taken during the months of January through May, from 1987 through 1990 at the P11C site are shown in Figure 1. Similar data from a site in which the surface 0.15 m of soil has been removed are presented in Figure 2. A summary of soil solution Se concentrations from the five monitored profiles from the P11 study area is provided in Figure 3. In this figure, soil solution Se concentrations within a given profile have been averaged over a period of two characteristic months within a given wet season, and normalized to the profile averaged concentration of the first year. This provides a convenient approach to discerning relative changes in the soil solution Se inventories over time in profiles of widely varying Se concentrations. Soil solution Se in all of these profiles occurred primarily as Se(VI), with usually minor amounts of Se(IV) (<10%). Although the volumetric soil water content profiles were not identical from year to year, their differences are judged to be minor based upon a combination of data from gravimetric moisture content determinations, neutron probe readings, and tensiometer data. Concentration changes, therefore, are interpreted as reflecting solution inventory changes. Nonuniformities in the soluble Se depth distributions (e.g. Fig. 1) make interpretation of these data difficult. However, time trends in Se concentrations obtained by averaging data within particular profiles permit detection of general patterns, especially when several different profiles are simultaneously evaluated in this manner. Thus, several general observations concerning the soil solution Se inventory at this upland site can be made, and are discussed below.

The major changes in observed Se concentrations occurred during the first 2 years of monitoring, while the most recent (1990) data show overall little difference from the preceding year. The three unexcavated upland plots exhibited 37% to 129% increases in average solution Se from 1987 to 1989. Incomplete sample collections during 1990 prevented meaningful averaging for two of these sites. However, the available data are indicative of relatively minor changes during the 1989 to 1990 period. From 1987 to 1989, average soil solution Se concentrations in the 0.15 m and 0.30 m excavated sites increased by 108% and 320%, respectively. No significant changes in profile-averaged Se concentrations were observed between the 1989 and 1990 data from these sites.

The increase in solution Se concentrations cannot be explained from external inputs, either from above or from below the sampling zone. Although downward leaching of Se from the soil surface by rainfall will provide some inputs into the strata monitored by the soil solution samplers, data from the excavated sites indicate that such a process appears inadequate for explaining the magnitude of the concentration changes. In the two excavated sites, where the most seleniferous surface soils have been completely removed, even greater increases in the profile-averaged Se concentrations were measured through the 1987-1989 (e.g. Figure 2). It also should be noted that increases in these soil solution profile Se concentrations can not be explained by Se inflows from the shallow water table. As is evident from Figure 1, Se concentrations in soil solutions at depths below 1 m are too low to provide significant relative changes within the upper soil profile over the monitored history (unless implausibly high flow rates, ranging from 3 to 50 m y $^{-1}$, were assumed).

The lack of significant changes in profile-averaged solution Se concentrations in these plots during the most recent samplings is not likely to be attributable to reaching solubility limits for any particular Se(VI) salt. The approximately stable average solution Se concentrations within each profile during the 1989 to 1990 often was associated with simultaneously occurring local concentration increases and decreases within particular sites (Figures 2 and 3). Individual soil solution samplers have continued provide time trends of pore waters with steadily increasing Se concentrations. In these cases other solution samplers within the same profile have provided pore waters with declining Se concentrations so that profile-averaged Se concentrations remained relatively unchanged. In light of the work of Brown and Carter (1969) and Elrashidi et al. (1987) which indicated that $BaSeO_4$ may control solubilities of Se(VI) in soils, this possibility was investigated.

Preliminary analyses for Ba in highly seleniferous Reservoir soil solutions indicate that these concentrations are at least an order of magnitude too low relative to the $BaSeO_4$ solubility. However, as noted by Stumm and Morgan (1981, chapter 5) and Sposito (1984, chapter 4), such comparisons with solubility products may be misleading.

The general time trends of initially rapid increases in the soil solution Se concentrations, followed by more recent minor fluctuations is consistent with the expected behavior of a seleniferous soil which has experienced a transition from reducing to oxidizing conditions. Although flooding of this upland site last occurred approximately 1 y prior to the initiation of this study, it is conceivable that the soil solution Se concentration increases observed during 1987 to 1989 represented the end of a period in which rapid release of a portion of the total Se inventory took place during a return to a more oxidizing soil environment. The present, relatively stable, high concentrations of Se in the soil solution do not appear to represent reaching of a solubility limit with respect to Se(VI) in solution.

Most of the increase in the soil solution Se concentration profiles appear to represent the release of Se from the *adsorbed, carbonate,* and *SOM* fractions discussed in section 3.2. The dynamic nature of the soluble Se fraction evident from the soil solution sampler data, as well as the presence of a much larger proportion of the Se which is currently not freely mobile in the soil solution, have motivated the detailed analyses of these other pools of upland soil Se at the Reservoir. The result of these investigations are discussed in the following section.

3.2 Distribution of Se in soil fractions

The distribution of Se in soil fractions at the surface (0 to 0.1 m) of the P11C site, according to the sequential extraction procedure described earlier, is presented in Table 1. Important elements (Ca, Mg, Al, Fe, Mn, Si, and S) that were released with each extraction are also presented in Table 1 so that the selectivity of each extraction can be evaluated.

The Se extracted by *KCl* (*soluble* Se) was found to be comprised of 77% Se(VI) and 23% Se(IV), consistent with the higher solubility of the former and the greater tendency for Se(IV) adsorption by soils. The large amounts of *KCl*-extracted Ca and S (virtually all SO_4^{2-}) indicate the dissolution of evaporite minerals such as gypsum. Other Reservoir soils that have extensive salt crusts have been found to contain *KCl*-extractable Se at levels greater than 7 mg kg^{-1} soil, and Se(VI) and Se(IV) at about the same relative concentrations as reported above for site P11C. The potential for precipitating gypsum to remove Se(VI) and Se(IV) from solution by occlusion and/or coprecipitation reactions is suggested from results of laboratory investigations (D. Suarez, U.S. Salinity Laboratory, U.S.D.A.-A.R.S., Riverside, CA, personal communication).

The *P8*-extracted Se pool (designated as *adsorbed* Se) was comprised predominantly of Se(IV) (65%), which reflects the greater tendency of Se(IV) to be adsorbed by charged surfaces. The negligible amounts of S in the *P8* extract indicates that the preceeding *KCl* extraction was effective in removing most of the soluble sulfate salts. The large amount of Se(VI) observed in the adsorbed pool was surprising given that it is not strongly sorbed by alkaline soils (Neal and Sposito, 1989), and that, according to the small quantity of S in the *P8* extract, a minor amount of evaporite salts were dissolved. The source of the *P8*-extracted Se(VI) is currently being investigated.

In the *OAc/P8* extraction that removes Se associated with *carbonates* (*P8* was used to desorb Se(IV) that readsorbs significantly at pH 5 during *OAc* extraction), the relative proportion of Se(IV) and Se(VI) was found to be similar to that reported for the *adsorbed* pool. Evidence for the removal of Se(IV) and, to a lesser degree, Se(VI) from solutions in which calcite is precipitating has been observed in laboratory investigations (Lipton, 1990; Suarez, personal communication). The substantial amount of Se(VI) in the *carbonate* fraction of the P11C soil was unlike that reported for two irrigated Western San Joaquin Valley agricultural soils by Lipton (1990) in which Se(IV) accounted for virtually the entire *carbonate* Se pool. This disparity is possibly a result of the higher Se(VI) content of the drainage waters that entered into Kesterson ($\approx$ 300 μg L^{-1}), relative to the concentrations of

Se(VI) in surface soils of irrigated agricultural fields, which could have resulted in greater amounts of Se(VI) being retained with precipitating carbonates.. Interestingly, with depth in the P11C soil profile, the proportion of *carbonate*--associated Se(VI) decreased to levels that more closely approached that observed in the surface soils of the agricultural fields.

Clearly, the largest amount of soil Se, more than 4 mg kg^{-1} soil, was extracted by *NaOCl*. The ability of this SOM extractant to minimize the dissolution of hydroxides and aluminosilicates while oxidizing organic C (>95% removed) is reflected in the small levels of *NaOCl* -extractable Fe, Al, Mn, and Si, especially when the amounts are compared to that removed by the subsequent extractions (Table 1). The pool of Se solubilized by *NaOCl*, as described in section 2.2, is considered to include forms of Se that are closely associated with the SOM (*SOM*-Se); organic Se, Se(IV) "protected" by a coating of SOM, and elemental Se whose production depends on the microbial respiration of organic compounds (Doran, 1982). A more detailed description of *SOM*-Se in Kesterson sediments, using a combination of methods developed by Weres et al. (1990) and Lipton (1990), will be presented in a future paper.

The Se associated with refractory inorganic soil constituents (*ER-Ox* + *Am-Ox* + *Cryst-Ox* + *Alk-sol Al/Si*) was found to comprise an insignificant proportion of the total soil Se (<2%). (The recovery of total soil Se by the sequential extraction procedure was 95.0% for the 0 to 0.1 m depth, and was generally 90% to 110% for all other depths.) Trivial amounts of Se in the refractory inorganic soil constituents were also observed throughout the rest of the profile.

It can be concluded from the sequential extraction procedure, therefore, that virtually all of the soil Se inventory was accounted for in the *soluble, adsorbed, carbonate*, and *SOM* fractions. This observation was distinct from investigations of surface California agricultural soils in which refractory inorganic pools (hydroxides and amorphous aluminosilicates) have been reported to play a prominent role in the retention of soil Se (Chao and Sanzalone, 1989; Lipton, 1990). This disparity is probably a consequence of the forms of the Se source as the majority of the Se was introduced to Kesterson via drainage waters in its most mobile form, Se(VI), while soil Se in the agricultural soils was derived from refractory Se-bearing sulfidic minerals originating from marine sediments. It is conceivable that over time some of the Se in the Reservoir soils may be incorporated into refractory inorganic soil constituents although this possibility is not likely to play an important role in the mitigation of Se pollution in the short term. The kinetics of Se transformations in soils, however, have not been elucidated for the wide range of expected conditions at the Reservoir.

The depth distribution of the major Se fractions at site P11C is illustrated in Figure 4. Total extractable Se is presented along with *soluble, soluble* + *adsorbed, soluble* + *adsorbed* + *carbonate*, and *SOM* pools of Se so that the relative importance of the soil fractions can be visualized throughout the profile. The general decrease in the quantities of Se with depth reflects the practice by which Se was introduced into this site, i.e. from surface applications of drainage waters, and the capacity of the Kesterson soil environment to immobilize large amounts of Se, most notably in the SOM. In the top 0.2 m, more than 70% of the total Se inventory was observed to be associated with the *SOM* fraction, highlighting the importance of the SOM in the biogeochemistry of Se. The relative significance of *SOM*-Se at about 0.5 m depth, however, declined markedly (to about 20%) as the *soluble* and *adsorbed* pools of Se increased proportionally. Interestingly, this trend was reversed deeper in the profile (0.7 to 1.0 m) as Se in the *soluble* and *adsorbed* pools were found in small amounts while *SOM*-Se was observed to reach a second maximum of almost 1.0 mg kg^{-1} soil. As previously noted, this portion of the profile corresponds to the region influenced by the annual maximum height of the water table. Preliminary investigations, using a combination of methods developed by Weres et al. (1989) and Lipton (1990), have indicated that the *SOM*-Se at the 0.7 to 1.0 m depth was largely elemental Se while at the surface it was primarily organic Se and SOM-associated Se(IV) (data to be presented in a future paper). This apparent increase in the percentage of elemental Se relative to the total Se inventory with depth and may have resulted from more reducing conditions at the fluctuating water table.

The presence of Se in *soluble, adsorbed, carbonate,* and *SOM* fractions, and its relative absence in refractory inorganic soil constituents, reflects the manner by which Se entered and was recycled within the Kesterson Reservoir environment. The potential for Se to be mobilized by common cultural and pedogenic processes (e.g. oxidation of SOM by tillage and drying, dissolution of carbonates and other evaporite minerals by acidification from plant roots and fertilizers, and displacement of adsorbed Se by phosphate fertilizers), suggests that the distribution of Se within Reservoir soils will change over time. On the other hand, presently only a small fraction of the Se at Kesterson is in solution, even after several years of exposure to oxidizing conditions. This illustrates the complexity of the behavior of Se in natural soil systems, and suggests that predictions of future behavior are premature.

4. ACKNOWLEDGEMENTS

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Table 1. Total Se, Se(IV), and some important elements removed by the sequential extraction of site P11C (0 to 0.1 m).

Soil Fractions Extractant(s)	Se$_T$	Se(IV)	Ca	Mg	Al	Fe	Mn	Si	S
	----μg/kg*----		-------------------------mg/kg*-------------------------						
Soluble:									
KCl	298	68	3,738	621.7	3.6	< 0.5	< 0.5	31.8	1,778
Adsorbed:									
P8	417	269	100.9	28.8	< 0.5	1.9	< 0.5	63.8	22.5
Carbonate:									
OAc	124	80	1,763	68.9	12.7	0.8	4.1	99.4	37.8
P8	104	58	1.0	< 0.5	4.4	5.3	< 0.5	61.0	< 0.5
SOM:									
NaOCl	4,341	NA	41.3	19.8	5.2	< 0.5	< 0.5	61.1	31.7
ER-Oxide:									
NH₂OH	< 4	NA	29.4	38.2	77.0	156.0	1.7	168.4	NA
KOH	8	NA	< 0.5	< 0.5	52.8	2.0	< 0.5	510.9	NA
Am-Oxide:									
NH₂OH/HCl	14	NA	110.4	331.9	980.7	1,059	7.0	951.7	NA
KOH	10	NA	< 0.5	< 0.5	10.0	< 0.5	< 0.5	1,749	NA
Cryst-Oxide:									
HCl	5	NA	477.3	4,069	10,049	18,476	111.8	924.4	NA
Alk-sol Al/Si:									
NaOH-1	38	NA	< 0.5	3.5	342.4	11.0	< 0.5	1,972	NA
NaOH-2	< 4	NA	< 0.5	< 0.5	203.8	6.0	< 0.5	1,007	NA
NaOH-3	< 4	NA	< 0.5	< 0.5	143.6	13.9	< 0.5	28,623	NA

* kg of air-dried soil.
Data are means of duplicate samples with relative % differences usually < 10% and always < 30%.
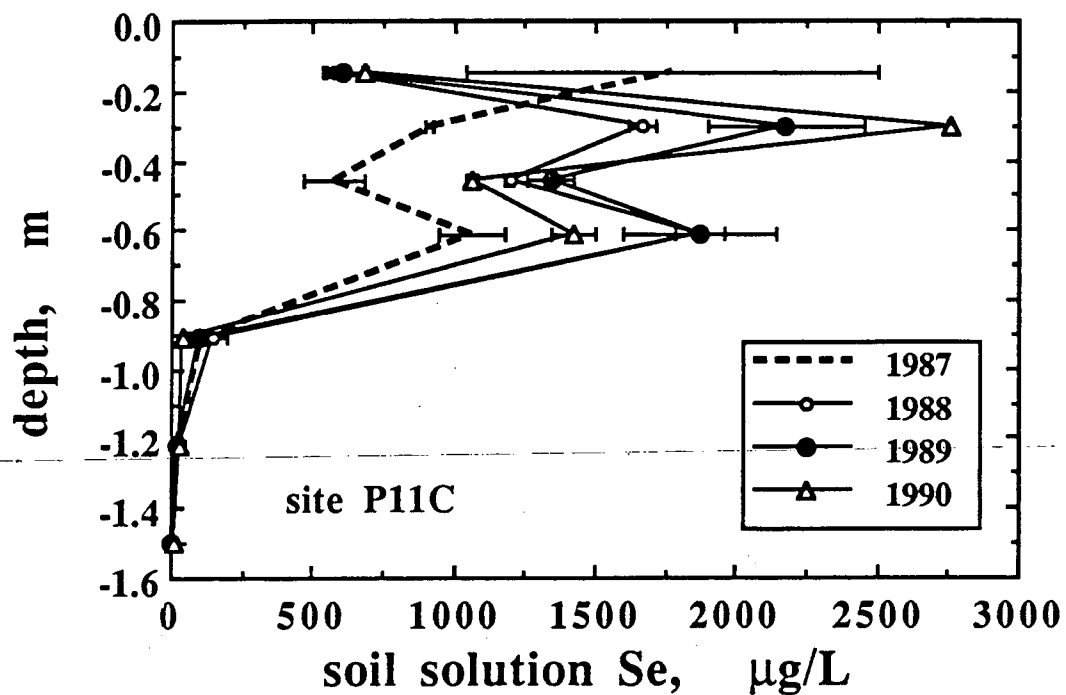NA: not analyzed.

Fig. 1. Soil solution Se concentrations profiles at site P11C, from 1987 to 1990.
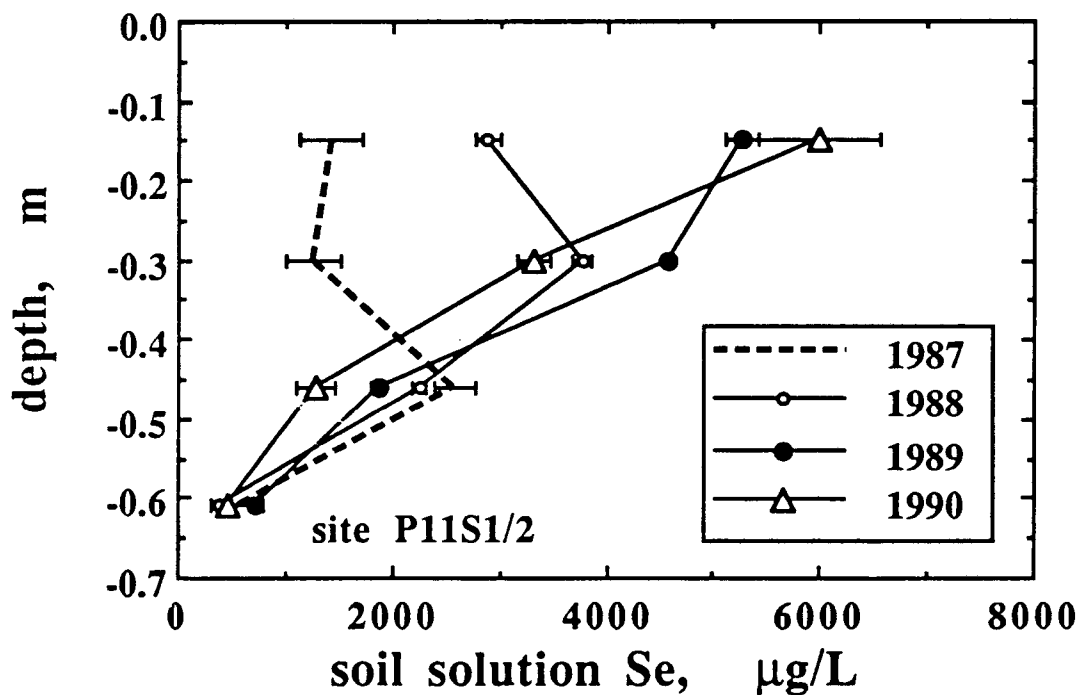

Fig. 2. Soil solution Se concentration profiles from site P11S1/2, which has had the original 0.15 m of seleniferous surface soil removed. Depths are relative to the current (excavated) soil surface.
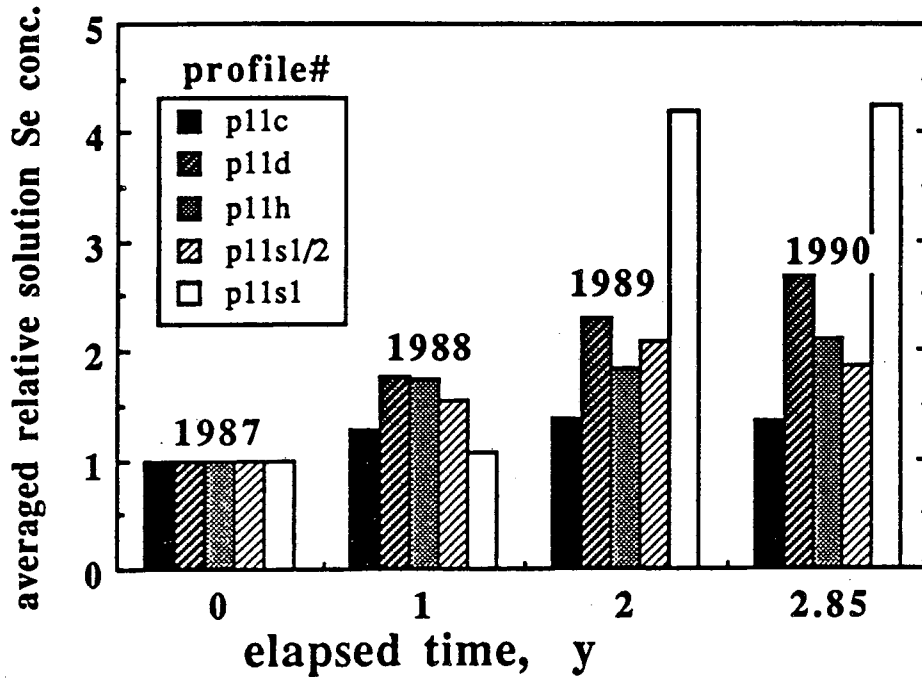
Fig. 3. Summary of profile-averaged *relative* concentrations of soil solution Se over time. The profile-averaged *relative* Se concentrations have been normalized to the initial (1987) profile averaged Se concentrations in the respective sites.
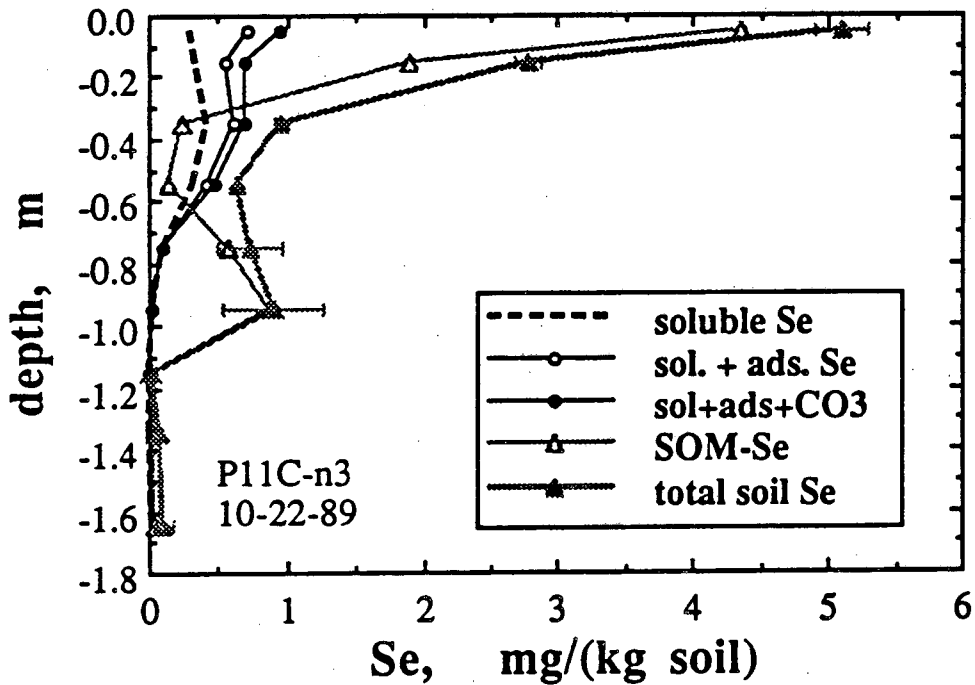

Fig. 4. Depth profile of soil-Se concentrations at the P11 study site.

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