MASTER

Chemical¹ Treatments of Soil to Decrease
Radiostrontium Leachability¹

Brian P. Spalding²

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²Research Associate, Environmental Sciences Division ORNL, P.O. Box X, Oak Ridge, TN 37830.

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ABSTRACT

The ready leachability of radiostrontium from radioactive waste is one of the most salient problems with shallow-land burial as a disposal method. The continuous leaching of buried waste at the Oak Ridge National Laboratory, for periods up to thirty years, has led to contamination of significant volumes of soil with ^{90}Sr . The goal of the present investigation was to evaluate methods to effect the in situ fixation or decrease the leachability of ^{90}Sr from soil. Small columns of three soils, collected from the solid waste disposal areas at ORNL, were labelled with ^{85}Sr as a convenient tracer for ^{90}Sr . After this labelling but prior to leaching, the soil columns were percolated with equivalent amounts of sodium salt solutions of hydroxide, fluoride, carbonate, phosphate, silicate, or aluminate. Leaching was then initiated with 0.1 N CaCl_2 (calcium chloride), and fractions of the leachate were analyzed for ^{85}Sr . The CaCl_2 solution was selected to qualitatively simulate groundwater which contains Ca as the dominant dissolved cation. With two soils, which were high in indigenous exchangeable Ca, only 30 to 35% of the ^{85}Sr could be leached from the carbonate-treated columns. Presumably, the ^{85}Sr was coprecipitated with the nascent CaCO_3 formed during this treatment. In contrast, greater than 98% of the ^{85}Sr was readily leached from all untreated soils. Other anions fixed variable but generally less ^{85}Sr than the carbonate treatment. Thus, sodium carbonate appears to have potential application to immobilize ^{90}Sr in situ in contaminated soil.

Additional Index Words: strontium fixation, sodium carbonate soil treatment, ^{90}Sr immobilization.

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3 The disposal of low-level radioactive wastes by shallow land burial
4 can pose potential problems due to the mobility of the long-lived fission
5 product ^{90}Sr (Francis, 1978). The close chemical similarity of Sr to Ca,
6 the dominant dissolved and mobile cation in most groundwaters, can
7 facilitate the leaching of ^{90}Sr . Leaching of buried waste at the Oak
8 Ridge National Laboratory has led to ^{90}Sr contamination of soils in and
9 around disposal areas. It would be desirable in the management of these
10 contaminated soils, as well as in the management of the waste itself, to
11 have a method to fix or immobilize radiostrontium in soil. The present
12 study was initiated to evaluate the in situ formation of insoluble calcium
13 phases with which ^{90}Sr would coprecipitate. In addition, if such phases
14 could be formed in situ, it was also desired to know how leachable or how
15 well fixed the ^{90}Sr in these phases would be. Sodium salts of anions,
16 including carbonate, fluoride, silicate, aluminate, and phosphate, were
17 selected since all of these form quite insoluble Ca salts. The hydroxide
18 was also studied because elevated soil pH can lead to enhanced radiostrontium
19 adsorption via enhanced cation exchange selectivity (Spalding, 1980).
20 The leachability of adsorbed radiostrontium induced by hydroxide has not
21 been examined, and other more stable phases, such as the carbonate, are
22 likely to form with aging of hydroxide-treated soils.

23 Several previous investigations have examined some of these precipitated
24 phases in soil, although a systematic, comprehensive study of all of these
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1 phases has not been attempted previously. Gregers-Hansen (1964) achieved
2 considerable Sr fixation in soil via heat treatment and, to a smaller
3 extent, via phosphate addition. Phosphate fertilization of soil does
4 lead to decreased plant uptake of radiostrontium through immobilization
5 in calcium phosphates (Anderson, 1971). Sodium carbonate has been used
6 to reduce plant uptake of radiostrontium from surface soil (Menzel et al.,
7 1968); in this case, sodium carbonate was not employed to directly
8 immobilize radiostrontium in CaCO_3 but rather to restrict rooting depth
9 of field crops following deep plowing of contaminated surface soil with
10 the alkaline carbonate. Mokady and Gal (1964) reported the fixation of
11 radiostrontium by lime in soil due to isomorphic exchange of Sr with the
12 indigenous CaCO_3 in soil using a SrCl_2 leaching solution; although this
13 fixation mechanism has potential, the use of Sr as the leaching cation
14 rather than Ca makes extrapolation of their findings most difficult to
15 typical environmental conditions where Ca dominates the solution phase.
16 Flocker and Fuller (1956) showed contrasting evidence in that ^{89}Sr was
17 very slow in isomorphic exchange with CaCO_3 phases in calcareous soils.
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MATERIALS AND METHODS

The collection, preparation, and characterization of the three soils used in this study have been described previously (Spalding, 1980).

These soil characteristics are summarized in Table 1. Water was added in 1-ml increments, followed by shaking and mixing, to 150 g of air-dried (<5% moisture, dry-weight basis) soil contained in a 500-ml glass bottle; this wetting procedure was continued until sand-sized soil aggregates were formed and dust-free air was observed in the bottle. Wetting of soils to this moisture state produced an aggregate size distribution which yielded facile and reproducible leaching patterns from packed soil columns. The final moisture contents of soils BG4U, BG6U, and BG6N were 17.8, 16.5, and 13.3%, respectively, determined by drying at 100°C to constant weight. Nine grams of this moist soil were poured, with tapping, into 1-cm-diameter by 10-cm-long glass-barrel Econocolumns (Biorad Laboratories, Inc.). The soils subsided to within 0.5 cm of the top edge of the glass barrel, achieving calculated packing densities of 0.99, 1.01, and 1.05 g/cm³, respectively, for the three soils.

To the top of each column was pipetted 1.00 ml of a stock solution of carrier-free ⁸⁵Sr (approximately 37 KBq/ml) in tapwater. The columns were allowed to age for 18 h before treating with one of six solutions. Solutions of NaOH, NaF, Na₂CO₃, Na₃PO₄·12 H₂O, Na₂SiO₃·9 H₂O, and NaAlO₃ were prepared to calculated Na concentrations of 1.56 N; actual Na concentrations, determined by atomic absorption of diluted solutions, were found to be 1.57, 1.57, 1.62, 1.70, 1.37, and 1.37, respectively. Two

1 milliliters of a chemical solution or, for controls, tapwater were pipetted
2 on the top of a soil column and allowed to infiltrate by gravity; no
3 leachate was produced by this treatment since the soils were still below
4 their water-holding capacities. Duplicate columns were prepared for each
5 chemical and control for each soil. After aging 18 h, leaching was
6 commenced by pipetting 5 ml of 0.1 N CaCl_2 on each column twice daily.
7 Generally, 12 to 16 h were required to percolate this 10 ml of leaching
8 solution through each column. After 120 ml of total leachate were col-
9 lected, the eluting solution was changed to 0.1 N HCl . Elution was then
10 continued until ^{85}Sr activity could no longer be detected in the eluant.

11 Activity of ^{85}Sr in each 10 ml eluant fraction, contained in 25 ml
12 plastic scintillation vials, was determined by counting with a well-type
13 NaI(Tl) gamma scintillation detector attached to a multichannel analyzer.
14 Five-minute counting times were employed and were generally adequate to
15 achieve an uncertainty in the count rate of less than 0.1%. Activity of
16 each fraction was expressed as a percent of the total activity applied to
17 a column. The pH of each fraction was also determined with a pH meter
18 using a combination reference-glass electrode.

RESULTS AND DISCUSSION

Chemical treatments effected two basic changes in the elution of ^{85}Sr from the soil columns. Firstly, there was an increase in the volume of 0.1 N CaCl_2 required to leach the largest concentration or peak of activity. Secondly, there was often substantial reduction in the fraction of the total activity which could be leached following chemical treatments. Considering the surface soil, BG4U (Fig. 1), approximately 26 ml were required to leach one-half the ^{85}Sr in the untreated soil, yielding a calculated column retention capacity of 28.8 $\mu\text{Ci/g}$ (Table 2). With the fluoride and carbonate treatments, the volume required to elute maximum activity with 0.1 N CaCl_2 yielded increased column retention capacities (Table 2). More importantly, although 75% of the ^{85}Sr could be leached by 100 ml of 0.1 N CaCl_2 , only 60 and 35% of the ^{85}Sr could be leached by 120 ml from the fluoride- and carbonate-treated soils, respectively. In addition, the slopes of the elution curves or the rates of leaching of ^{85}Sr from the fluoride- and carbonate-treated columns had decreased substantially by 120 ml, indicating that numerous additional column volumes of 0.1 N CaCl_2 would be required to leach out the ^{85}Sr . In the case of the carbonate, this rate of leaching was so slow (Fig. 1) that the remaining ^{85}Sr could reasonably be considered fixed or unleachable.

The form of this fixed ^{85}Sr was most probably a coprecipitant or solid solution of ^{85}Sr with or in CaCO_3 and CaF_2 . Such precipitated forms of Ca would be expected to readily dissolve in acid; changing the

1 eluting solution to 0.1 N HCl led to the facile leaching of this fixed or
2 precipitated ^{85}Sr as shown in Fig. 1. The soil columns of soil BG4U,
3 which were treated with silicate, phosphate, aluminate, and hydroxide,
4 could not be leached with 0.1 N CaCl_2 due to column plugging by these
5 strongly alkaline chemicals, caused by dispersion of aggregates and soil
6 organic matter. Such flow stoppages, induced by these chemicals, were
7 not observed with the two subsoils with organic matter contents of less
8 than 0.1%.

9 As shown in Fig. 2, the neutral subsoil, BG6N, exhibited almost
10 identical elution patterns to BG4U for the control and carbonate treatments.
11 The fluoride treatment of this soil, however, immobilized less ^{85}Sr than
12 the surface soil (18 versus 40%, respectively). This soil did allow the
13 comparison of all the $\text{Ca}/^{85}\text{Sr}$ immobilizing treatments. As discussed
14 above, there are several ways in which these treatments should be compared.
15 These are by elution volumes of maximum leachable activity, by total
16 amounts of leachable ^{85}Sr by the 120 ml of 0.1 N CaCl_2 , and by rates of
17 elution or slopes of the lines just before the leachate is changed to
18 0.1 N HCl. To estimate the first two of these characteristics, the
19 elution curves were fitted to a general Gaussian equation:
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$$21 \quad \text{Fraction } ^{85}\text{Sr}/10 \text{ ml} = A \exp\{-(B - \text{elution volume}) C\}^2, \quad (1)$$

22 where the constants A, B, and C are estimates of the percent activity in
23 the maximum or peak fraction, the elution volume of this maximum fraction,
24 and a measurement of dispersion or width of the elution peak, respectively.
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1 These estimates of the three constants for each soil and chemical treatment
2 were determined using a nonlinear least-squares regression computer
3 program (SAS Institute, 1979). The model used actually explained an
4 average of 92% of the variation in predicted elution in all cases.

5 It can be seen from Table 2 that all treatments raised the peak
6 elution volume, B, by factors ranging from 1.2 to 7.5 and, consequently,
7 led to increases in column retention capacities. All of the treatments
8 resulted in elevating the pH of the initial leachate (Table 2). Such pH
9 increases result in increases in pH-dependent charge on the soil particles
10 as well as in a neutralization of exchangeable acidity. Since the newly
11 created exchange sites, effected by these alkaline treatments, would
12 contain Na as the charge-balancing cation, they will also exhibit selectivity
13 for exchange by Ca and ^{85}Sr from the leaching solution (Spalding, 1980).
14 This effect is clearly demonstrated in the case of soil BG6U, the acid
15 (pH 4.2) subsoil; all treatments effected at least a four-fold increase
16 in the soil retention capacities. The greater exchangeable acidity of
17 this acid soil would lead to greater Na saturation following chemical
18 treatment and, hence, lead to greater increases in Ca or ^{85}Sr retardation.
19 Notably, the sum of exchangeable bases for soils BG4U, BG6N, and BG6U
20 were 16.3, 24.6, and 6.7 meq/100 g (Table 1), which roughly correspond to
21 the calculated retention capacities for untreated soils (28.8, 29.8, and
22 7.0 meq/100 g, respectively, Table 2). The two other near-neutral soils
23 exhibited less increase in their retention capacities.

1 This acid subsoil was not, in general, able to immobilize as much
2 ^{85}Sr in response to the chemical treatments as were the other two soils
3 (Fig. 3). One interpretation of this behavior can be seen from the
4 nature of the indigenous cations on the soils (Table 1). Whereas soils
5 BG4U and BG6N contained 15.5 and 24.2 meq/100 g indigenous exchangeable
6 Ca and Mg, soil BG6U contained only 6.5 meq/100 g. There would be,
7 therefore, less Ca and Mg present in this soil to form the precipitated
8 phases with the various anions. In addition, much of the added anion
9 would be consumed in neutralizing the exchangeable acidity in this acid
10 soil. The exchangeable acidity (to pH 8) of soil BG6U would require
11 1.55 meq or 50% of the added alkalinity of each chemical. However, BG4U
12 and BG6N, with their lower exchangeable acidities would require only 19
13 and 16%, respectively, of the applied alkalinity. Apparently, both the
14 consumption of precipitating anions and the smaller amounts of Ca and Mg
15 indigenously present led to the decreased amount of precipitated phases
16 in soil BG6U.

17 The actual amount of ^{85}Sr , which can be precipitated in a given
18 soil, can be considerably varied by operating parameters. The addition
19 of more chemical for acid soils, the addition of more Ca in those soils
20 low in indigenous amounts, or the use of longer columns would all be
21 expected to lead to greater fixation of ^{85}Sr . Factors which cannot be
22 manipulated by operating variables and, hence, are more fundamental from
23 the point of view of the in situ immobilization of ^{90}Sr in soil, are the
24 rates of isomorphic exchange of the precipitated activity with the Ca in
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1 the leaching solution or groundwater. These rates of the isomorphic
2 exchange of the coprecipitated ^{85}Sr activities can be calculated, in
3 comparative units, from the slopes of the elution lines in the final
4 fractions of leachate. The average percent activity, eluted in the final
5 three fractions with 0.1 *N* CaCl_2 , is presented in Table 2. Notably, the
6 carbonate phase exhibited the lowest isomorphic exchange rates of any of
7 the precipitated phases. Even in the case of soil BG6U, where the
8 carbonate treatment immobilized the least amount of activity after 120 ml
9 of leaching (Fig. 3), that immobilized fraction was much slower to
10 exchange than those of other treatments. The pH's of all the final
11 leaches for all chemically treated columns were the same; the mean of all
12 32 treated columns was $7.2 \pm 0.4(1\sigma)$. This condition would obviate pH
13 differences among treatments affecting the solubilities of precipitated
14 phases.

15 The 0.1 *N* CaCl_2 , employed as the leaching solution in this study,
16 simulates groundwater only in the sense that Ca is the dominant dissolved
17 cation. The concentration of Ca in a typical groundwater would be at
18 least an order of magnitude lower and would have bicarbonate rather than
19 chloride as the anion. The higher concentration of Ca was employed here
20 to speed up the elution from these columns; three weeks were required to
21 obtain the total leachate volume in this study, while a tenfold longer
22 time would have been required to achieve the equivalent amount of leaching
23 using 0.01 *N* CaCl_2 . Some precipitated phase likely formed during the
24 initial leaching with 0.1 *N* CaCl_2 , before excess anion was leached out,
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1 which would not have formed had a lower Ca concentration been employed.

2 In terms of developing a technique for the in situ fixation of ^{90}Sr ,
3 however, this does not pose too much difficulty since supplemental Ca
4 could be added after chemical treatment if the indigenous Ca groundwater
5 concentration were inadequate to effect the desired level of fixation.

6 On the other hand, since the diffusion coefficient of ^{90}Sr in soil
7 increases exponentially with increasing Ca concentration in the soil
8 solution (Prokhorov and Frid, 1965), the leachability of ^{85}Sr from the
9 various precipitated phases by 0.1 N CaCl_2 would be faster than that with
10 more realistic Ca concentrations. Thus the 0.1 N CaCl_2 used in this
11 study provides a more rigorous test of the fixation of Sr in the various
12 precipitated phases, assuming that diffusion limits isotopic and isomorphic
13 exchange between solution and solid.

14 Although the solubilities and coprecipitations with various $\text{Ca}(\text{Sr})$
15 phases have been investigated intensively in the course of developing
16 radiochemical separation techniques (Sunderman and Townley, 1960), the
17 ability of these phases to form in situ in soil has not been investigated
18 systematically previously. Although solubility products did provide a
19 basis for selecting the various precipitated phases for investigation,
20 they do not provide a basis to predict either the rates of precipitation
21 or the degree of isomorphic exchangeability of the precipitated activity
22 once formed. It would appear from this study that nascent $\text{Ca}(\text{Sr})\text{CO}_3$ can
23 be formed in soil in situ and lead to a marked reduction in the leachability
24 of radiostrontium. This conclusion is supported by the observations of
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1 Flocker and Fuller (1956) that CaCO_3 in calcareous soils was very poorly
2 available to plants because it would not isomorphically equilibrate in
3 several months with spiked ^{89}Sr used as a tracer for Ca. Other immobilized
4 phases in soil have been investigated even less, although it has been
5 known for some time that heavy application of phosphate can reduce plant
6 uptake of ^{90}Sr from soil (Anderson, 1971); presumably, this effect was
7 due to precipitation of some species of Ca phosphate. Notably, the
8 facile leachability of Sr by CaCl_2 in untreated soils provides a convenient
9 method to decontaminate a given soil rather than fix the radiostrontium
10 present. Such methods for decontaminating soils of radiostrontium have
11 been proposed previously (Lagerwerff and Kemper, 1975). In the case of
12 waste disposal, in situ fixation would seem more desirable since in situ
13 decontamination would require the thorough collection of leachate; otherwise,
14 accelerated contamination of groundwater would result.

CONCLUSIONS

The in situ treatment of soil with sodium carbonate can immobilize up to 70% of the radiostrontium present. In addition, the nascent coprecipitated $\text{Ca}(\text{Sr})\text{CO}_3$ was in very slow isomorphic exchange with Ca in the leaching solution. Other chemical treatments including silicate, aluminate, fluoride, phosphate, and hydroxide produced precipitated phases which, by comparison with the carbonate, were in more rapid isomorphic exchange with Ca in solution. In addition to producing insoluble phases in situ, all six chemical treatments retarded the leaching of that fraction of nonprecipitated radiostrontium by increasing the cation exchange capacity of the soil as well as the soil's selectivity for radiostrontium. Sodium carbonate treatment of soil appears to have potential for immobilization of radiostrontium in contaminated soils.

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Figure and Table Captions

Table 1. Characteristics of three soils derived from Conasauga shale.

Table 2. Characteristics of ^{85}Sr elution from soil columns.

Figure 1. Elution of ^{85}Sr from columns of soil BG4U with 0.1 N CaCl_2 following chemical treatments.

Figure 2. Elution of ^{85}Sr from columns of soil BG6N with 0.1 N CaCl_2 following chemical treatments.

Figure 3. Elution of ^{85}Sr from columns of soil BG6U with 0.1 N CaCl_2 following chemical treatments.

Table 1 — Characteristics of three soils derived from Conasauga shale.

Characteristic	Units	Soil		
		BG6U	BG6N	BG4U
Horizon	—	C	C	Ap
Organic matter	%	<0.1	<0.1	3.9
Fe ₂ O ₃	%	2.66	1.14	2.04
MnO ₂	%	0.64	0.22	0.09
Exchangeable acidity	meq/100 g	20.6	6.5	8.2
Exchangeable Ca	meq/100 g	2.4	17.2	12.6
Exchangeable Mg	meq/100 g	4.1	7.0	2.9
Exchangeable K	meq/100 g	0.1	0.2	0.8
Exchangeable Na	meq/100 g	0.1	0.2	0.0
Cation exchange capacity	meq/100 g	27.3	31.1	24.5
Exchangeable Sr	µeq/100 g	9	25	25
pH	—	4.4	7.3	6.5

Table 2 — Characteristics of ^{85}Sr elution from soil columns.

Soil	Chemical	Initial eluant pH	^{85}Sr elution model constants [†]			Retention capacity [‡]	Average ^{85}Sr eluted for 100-120 ml
			A	B	C		
			(fraction/10 ml)	(ml)	(ml ⁻¹)	(meq/100 g)	(%/10 ml)
BG4U	None	7.2	0.31	26	0.056	28.8	—
BG4U	CO_3^{2-}	9.4	0.07	32	0.054	36.9	0.43
BG4U	F^-	8.7	0.12	45	0.040	54.5	1.77
BG6N	None	6.7	0.35	27	0.066	29.8	—
BG6N	CO_3^{2-}	9.8	0.09	43	0.051	51.1	0.50
BG6N	F^-	9.2	0.21	45	0.047	53.7	1.20
BG6N	SiO_3^{2-}	11.4	0.10	59	0.028	72.4	3.15
BG6N	PO_4^{3-}	9.7	0.09	53	0.034	64.4	1.56
BG6N	AlO_2^-	11.2	0.12	50	0.034	60.4	2.00
BG6N	OH^-	12.7	0.12	62	0.028	76.4	3.48
BG6U	None	4.2	0.50	10	0.055	7.0	—
BG6U	CO_3^{2-}	9.5	0.35	41	0.075	46.8	0.59
BG6U	F^-	7.7	0.23	48	0.070	55.7	2.75
BG6U	SiO_3^{2-}	10.6	0.15	75	0.041	90.3	3.16
BG6U	PO_4^{3-}	7.6	0.33	46	0.082	53.2	1.45
BG6U	AlO_2^-	10.1	0.35	52	0.102	60.9	1.46
BG6U	OH^-	10.4	0.23	57	0.059	67.3	2.26

[†] Constants in Eq. [1].[‡] Retention capacity = (B — column void volume) × N of CaCl_2 /weight of soil (in hundreds of g).

NEUTRAL SURFACE SOIL - 67 % BASE SATURATION

Figure 1. Elution of ^{85}Sr from columns of soil BG4U with 0.1 N CaCl_2 following chemical treatments.

NEUTRAL SUBSOIL-79 % BASE SATURATION

Figure 2. Elution of ^{85}Sr from columns of soil BG6N with 0.1 N CaCl_2 following chemical treatments.

Figure 3. Elution of ^{85}Sr from columns of soil BG6U with 0.1 N CaCl_2 following chemical treatments.