
Anion Retention in Soil: Possible Application to Reduce Migration of Buried Technetium and Iodine

Development of a Field Test

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Prepared for
U.S. Nuclear Regulatory Commission

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ABSTRACT

Before testing the performance characteristics of andisols for retention of anions in the near-field environment of LLW disposal facilities it is necessary to locate one or more sufficiently extensive bodies of natural soil with the highest possible natural anion exchange capacity. For this purpose we developed a rugged, portable semiquantitative field test for anion exchange capacity based on short-term sorption of iodide by soil samples. We validated the iodide sorption field test against a well established quantitative laboratory test based on anion exchange of chloride and nitrate, then carried out an initial survey of volcanic terrain in northern California using the field test.

Based on our experience with the iodide sorption field test, we suggest these tentative guidelines for identifying probable anion exchange capacity in soil samples:

Iodide sorbed (meq/100 g soil)	Probable anion exchange capacity
< 0.1	Negligible
0.1-0.5	Low
0.5-1.0	Moderate
>1.0	High

In our northern California evaluation of the iodide sorption field test we found that:

- (i) All samples that sorbed iodide at levels ≥ 0.34 meq I/100 g soil in the field test showed anion exchange capacity > 0.04 meq Cl/100 g soil.
- (ii) The test showed no false negatives (cases where iodide sorption is zero and the sample shows anion exchange capacity).
- (iii) The test showed no false positives (cases where the test predicts anion exchange capacity that is not there).

The sample with the highest iodide sorption in the field test (0.91 meq I/100 g soil) also had the highest anion exchange capacity in laboratory validation by chloride-nitrate exchange (0.94 meq Cl/100 g soil at pH 5.8 with 0.001 N KCl equilibrating solution).

In an Appendix we describe an analytical procedure for titrating chloride in 1 N potassium nitrate using a Collove chloride titrator.

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ANION RETENTION IN SOIL: POSSIBLE APPLICATION TO REDUCE MIGRATION OF BURIED TECHNETIUM AND IODINE • DEVELOPMENT OF A FIELD TEST •

INTRODUCTION

On May 23, 1991, the U.S. Nuclear Regulatory Commission (NRC) held an informal one-day brainstorming session at Rockville, MD, to seek innovative means for containing radionuclides in Low Level Radioactive Waste (LLW) disposal facilities. The question before the group was "how could LLW disposal be done better?" Participants included NRC staff and research investigators from Brookhaven National Laboratory, Idaho National Engineering Laboratory, National Institute of Standards and Technology, Pacific Northwest Laboratory, Sandia National Laboratory, and the University of California, Berkeley.

Among the new ideas discussed was the possible use of a natural anion exchanger for conditioning the near-field environment around LLW disposal units to retard the migration of anionic radioisotopes such as iodine and technetium. Gu and Schulz (1991) reviewed the subject of anion exchange in soils, with special interest in these elements. Their review indicated that andisols, a soil-taxonomic group consisting mostly of volcanic soils, are the most likely source of substantial anion exchange capacity at pH near 6.

High anion exchange capacity soil would be placed around a LLW disposal facility to retain anions on the same principle that retains them in a column of anion exchange resin. That is, the solid phase (exchanger) carries numerous fixed sites of positive surface charge that give the material 'anion exchange capacity' and the ability to retain and exchange anions.

The terms 'sorption', 'ion exchange', and 'retention' all refer to the distribution of ions between exchanger and solution, and we should try to be clear about the meaning of these terms in our context:

'sorption' [PHYS CHEM] A general term used to encompass the processes of adsorption, absorption, desorption, ion exchange, ion exclusion, ion retardation, chemisorption, and dialysis.¹

'ion exchange' [PHYS CHEM] A chemical reaction in which mobile hydrated ions of a solid ion exchanger are exchanged, equivalent for equivalent, for ions of like charge in solution.¹

'Retention' of ions occurs during transport of solution through a porous exchanger that has fixed sites of opposite charge on its surface. The ions tend to be drawn to the solid surface, and this slows or stops their movement. Ions may be drawn with varying force to various

¹ McGraw-Hill Dictionary of Scientific and Technical Terms, Daniel N. Lapedes (Ed). McGraw-Hill Book Company, New York, 1976.

solid surfaces, so that differing degrees of attraction result in differing degrees of movement of the ions. Ion exchange in soil is probably the quickest element of a general sorption process.

STATEMENT OF THE PROBLEM

Data on the range and extent of active anion exchange capacity in natural soils, including andisols, are not available. Our problem is to locate precisely the range and extent of one or more large bodies of soil with unusually high anion exchange capacity. Evaluation of a large number of soils may be necessary to accomplish this. As laboratory tests of anion exchange capacity are time consuming and therefore expensive, our objective was to develop a rapid, semiquantitative field test for anion exchange capacity.

Our hypothesis in the field test is that if the anion exchange element of sorption is especially large in a given sample, and if our index anion is strongly selected, then a high level of sorption of the index anion will distinguish the sample from other samples with low or moderate anion exchange capacity.

BACKGROUND

Nature has been beneficent, but not evenhanded, in providing soil with the capacity to retain and exchange ions from soil solution. Ion exchange capacity in soil can serve such useful functions as retaining plant nutrients in surface soil and slowing the migration of ionic soil contaminants from toxic waste sites.

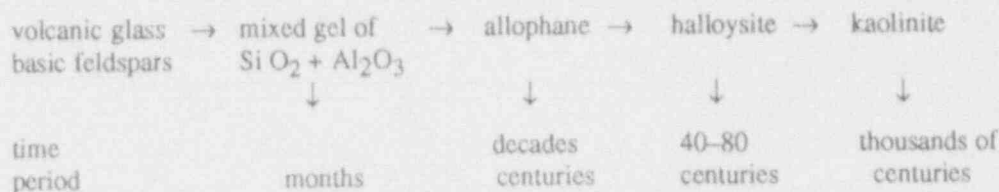
In the vast majority of soils cation exchange capacity is much greater and more stable than anion exchange capacity, due largely to permanent negative charge arising from isomorphic substitutions in clay minerals. Permanent *positive* charge in clay minerals is unusual (Tessens and SitiZaayah 1982). Anion exchange capacity usually arises from pH-dependent positive charge on surfaces of iron and aluminum oxides and hydroxides, where the positive charge develops only at low pH. Soils with significant pH dependent charge have been called 'variable-charge soils' (Barrow 1987).

Andisols² are variable-charge soils that can be exceptional with respect to anion exchange capacity because they commonly contain substantial amounts of allophane/imogolite, a clay-mineral precursor that may retain considerable positive surface charge at pH 6 (Gu and Schulz 1991). Andisols comprise the newest taxonomic unit in the classification system of the U.S. Soil Conservation Service (Soil Survey Staff 1990). A large percentage of these soils have formed in volcanic parent material, as described below. Surface horizons are typically dark brown, containing humified plant litter. Although much work has been done to describe and classify andisols, their anion exchange properties have not been measured and recorded in soil surveys. Furthermore, the presence of allophane/imogolite in soil, which may be recorded in soil surveys, does not necessarily confer high anion exchange capacity. Allophane/imogolite is itself a heterogeneous material,

²The term is spelled 'andosols' in some international usage. It is derived from a Japanese phrase meaning 'dark colored soils'.

consisting mainly of imperfectly crystallized aluminosilicates with notable variation in silicon/aluminum ratio. Furthermore, a variety of gel coatings may modify ion exchange properties in a particular soil (Robert and Terce 1989).

Allophane/imogolite is an intermediary mineral that may develop in soil profiles during the transformation of volcanic glass and basic feldspars to more weathered products. In a discussion of profiles of volcanic soils Mohr et al. (1972) refer to a time scale constructed by Besoain (1969) in which allophane/imogolite is present as an intermediate product:



Mohr et al. (1972, p. 411) point out that whether the reaction will proceed as indicated above depends on the fate of soluble reaction products:

If these are removed readily (rapid permeability), the reaction will be more complete than if the leaching process is slow. If leaching is slow it is even possible that allophane is not formed at all; if, for example, there is sufficient Mg (set free upon weathering of ferromagnesian minerals), montmorillonite may be formed.

The transition period in which allophane/imogolite can be abundant (ca 10,000 years), between mixed gel of $\text{SiO}_2 + \text{Al}_2\text{O}_3$ and mature clay mineral, is very long by most human standards. The rate and direction of soil development appears to depend on numerous factors including rainfall, soil permeability and topographic location.

A more detailed scheme of Wada (1985), but without a definite time scale, shows the development of allophane/imogolite in subsurface B, Bb, and Ab horizons in the early and middle stages of pedogenesis. In the late stage allophane/imogolite is replaced by halloysite, gibbsite, Al and Fe-humus, Fe oxide, and various 2:1 clay minerals.

Integrating this information, investigators might look for allophane/imogolite in subsurface horizons of volcanic soils that have developed for more than 200 and less than 10,000 years. Our purpose in this study was to develop a rapid field test for anion exchange and to begin to use the test for a survey of soils possibly having high levels of anion exchange capacity.

METHOD OF INVESTIGATION

Iodide sorption field test

To locate materials with high anion exchange capacity we developed a rapid field test using iodide sorption as an indicator of active anion exchange capacity. We chose iodide as our index anion because it is strongly sorbed and can be easily determined with a halide-specific electrode.

Field gear are rugged and portable, similar to that used for measuring soil pH in the field (Beckman Φ 11 pH meter and Orion electrodes 94-53 and 90-02). The dimensions of the battery-powered pH-millivolt meter are 5" \times 3" \times 1". The halide-specific electrode and reference electrode are mounted on a stand that fits into an ordinary backpack.

In preliminary experiments we used the halide-specific electrode (Orion model 94-53) to measure iodide sorption on Bio-Rad AG®1 Strong Anion Exchange Resin, for which the relative selectivity of counterions is given by the manufacturer (Bio-Rad Laboratories, 1985):

Counterion Relative Selectivity for AG 1 Resin

Counterion	Relative Selectivity for AG 1 Resin
OH ⁻	1.0
I ⁻	175
NO ₃ ⁻	65
Cl ⁻	22
F ⁻	1.6

The order of relative selectivities for ion exchangers is influenced to a large degree by the properties of the exchanging ions, such as their ionic radii (or hydrated radii) and their polarizability. The order of relative selectivities for soil colloids should be similar to that for AG 1 resin, and with similar magnitude. We developed the field test based on the short term sorption of strongly selected iodide by soil samples suspended in potassium iodide.

About 28 g (1 oz on a postal scale) of screened field moist soil was placed in a 250 ml wide-mouth plastic bottle (Nalgene) to which was added 130 ml (100 ml graduated cylinder filled to the brim) of potassium iodide solution (pH 5.8) containing 1 meq of iodide ($7.65 \times 10^{-3} N$). The bottle was capped tightly and shaken by hand several times (15–20 sec. each time). One to 18 hours later (depending on convenience) the supernatant solution was poured into a filter unit (Corning 25900-115) and vacuum-filtered with a hand-operated vacuum pump (Nalgene 54903-830). Iodide remaining in the filtrate was measured by the Orion 94-53 halide electrode coupled with a double-junction reference electrode (Orion 90-02) attached to a small, battery-powered meter (Beckman Φ 11 pH meter). Iodide sorbed by soil samples was calculated by difference.

Laboratory determination of anion exchange capacity

To verify the iodide sorption field test it was necessary to compare sorption results with anion exchange capacity determined by a well-established method. We adopted with modifications a frequently used method of measuring anion exchange capacity in allophanic clays, i.e., saturation of anion exchange sites with chloride at pH near 6, displacement of chloride by nitrate, and measurement of chloride in soil extracts (Perrott 1977, Gonzales-Batista et al. 1982, Theng et al. 1982, Clark and McBride 1984). Chloride and nitrate were chosen for anion exchange capacity measurements because, in the circumstances of the determination, anion exchange constitutes nearly the whole sorption process (Gebhardt and Coleman 1974).

Ten to 15 g of field moist soil was placed in a filter unit (Corning 25900-115) to which 50 ml of 2 *N* potassium chloride was added. After thorough mixing with a rubber policeman the < 0.2 μm solution was extracted under vacuum. This process replaced resident exchangeable anions with chloride. Four similar 50 ml rinses with 0.001 *N* potassium chloride (pH 5.8) further saturated anion exchange sites with chloride while simultaneously reducing the amount of chloride entrained in soil solution. Samples were then extracted with 115 ml of 1 *N* potassium nitrate.

A Ross® Sure-Flow combination electrode (Orion 8165) was used to measure pH of the final-rinse chloride extracts which ranged from 5.5 to 7.3, with a mean of 6.4 ± 0.75 ($n=15$).

Chloride entrained in soil solution after the final rinse was calculated from the weight of the entrained solution and its chloride concentration. Electrical conductivity ($\mu\text{S}/\text{cm}$) was converted to concentration (meq KCl/L) with the regression equation

$$(\mu\text{S}/\text{cm}) = 0.1459 (\text{meq KCl}/\text{L}) + 0.0073,$$

which spans the range 1–3 meq KCl/L. The mean electrical conductivity of final rinses (8/20/92) was 1.5 ± 0.3 meq Cl^-/L ($n=8$).

Chloride in the potassium nitrate extract was measured with a Cctlove chloride titrator (Aminco). Titrations were accurate and precise,³ with coefficients of variation $\leq 0.4\%$. Error for a complete anion exchange experiment depended mainly on care in weighing and transferring soil and solutions. Replicate experiments showed overall error ranging from 35% to 7%, depending mainly on operator care.

In preparation for field sampling we tested our methods with Aiken loam, Challenge clay loam, and Windy loam, three Sierra Nevada soils obtained from archives of the Department of Soil Science at Berkeley. In addition, we tested an unnamed soil from Shasta County that had been shown to contain 17% to 18% of allophane/imogolite by weight in a taxonomic study of andic soils (Southard and Southard 1989). Validation of the methods, however, depends primarily on results with freshly-collected field samples (See discussion on p. 13).

³ Appendix 2 describes measurement of chloride in 1 *N* potassium nitrate.

Field sampling in northern California

In May and July 1992 we travelled to Clear Lake (Lake County) and Mount Shasta (Shasta County), CA. From these areas of Pleistocene volcanic ash and lava deposition we collected 87 samples from 25 soil profiles developed in volcanic parent material (Fig.1)



Fig. 1 Sample Locations (See Appendix 1 for sampling coordinates)

In Lake County we were guided by the Lake County soil survey (Smith and Broderson 1989) to sites where we collected samples with soil-auger and shovel. In Shasta County U.S. Forest Service staff took us to backhoe pits used by the Forest Service in an ongoing study of andisols of the Shasta-Trinity National Forest. Samples were collected with a shovel after exposing fresh surfaces in the backhoe pits. In Lake County coordinates of sampling sites were read from soil survey maps. In Shasta County they were indicated by a Sony Global Positioning System receiver.

During the process of developing the field test we used it in the laboratory as well as in the field. Samples were stored at room temperature in double-walled zip-lock plastic bags to retain soil moisture. Mean gravimetric water content of the samples was $25\% \pm 6\%$ ($n=17$).

RESULTS

Iodide sorption field test

Table 1 (p. 7) includes both results of preliminary experiments that were necessary for methods development (Sierra Nevada soils) and results of our first efforts in testing soils in volcanic terrain (Lake County and Shasta County soils).

Of the Sierra Nevada soils, Aiken loam and Challenge clay loam are non-volcanic soils that had been stored in an air-dry condition. Windy loam is a volcanic soil that had been kept continuously moist in storage. Our first iodide sorption field tests (Table 1) showed iodide sorption by Windy loam, where we hoped to find it, but not by Aiken loam or Challenge clay loam. These results encouraged us to continue development of the iodide sorption field test.

Iodide sorption by the volcanic soils we tested ranged from 0 to 0.91 meq I⁻/100 g soil, with a mean of 0.34 ± 0.2 . Eighty-five percent of the samples sorbed between 0.1 and 0.7 meq I⁻/100 g moist soil.

TABLE 1
IODIDE SORPTION BY WHOLE SOILS FROM NORTHERN CALIFORNIA

Sample	Soil Type	Sample Depth cm	Iodide sorbed meq/100 g	Sample	Soil Type	Sample Depth cm	Iodide sorbed meq/100 g
SIERRA NEVADA SOILS				47	(Coonrod sandy loam)	0-17	0.24
1	Aiken loam	0-30	-0.02	48	(Coonrod sandy loam)	17-33	0.21
2	Challenge clay loam	0-30	-0.01	49	(Coonrod sandy loam)	33-43	0.35
3	Windy loam	40-60	0.46	50	(Coonrod sandy loam)	36-72	0.38
3	Windy loam	40-60	0.34	51	(Coonrod sandy loam)	72-86	0.45
LAKE COUNTY VOLCANIC SOILS				52	(Coonrod sandy loam)	88-107	0.45
4	Benridge-Konocti loam	0-30	0.20	53	(Coonrod sandy loam)	107-119	0.35
5	Benridge-Konocti loam	30-60	0.22	54	Depner sandy loam	0-30	0.56
6	Benridge-Konocti loam	60-90	0.25	55	Depner sandy loam	30-60	0.63
7	Benridge-Konocti loam	90-105	0.31	56	Depner sandy loam	30-60	0.56
8	Benridge-Konocti loam	0-30	0.23	57	Depner sandy loam	60-90	*0.59
9	Benridge-Konocti loam	0-30	0.46	58	Depner sandy loam	90-120	*0.52
10	Benridge-Konocti loam	0-30	0.47	59	Depner sandy loam	120-150	*0.63
11	Benridge-Konocti loam	30-60	0.44	60	(Edson sandy loam)	0-16	0.42
12	Benridge-Konocti loam	60-90	0.42	61	(Edson sandy loam)	16-60	0.66
13	Benridge-Konocti loam	90-120	0.42	62	(Edson sandy loam)	60-80	0.59
14	Benridge-Konocti loam	120-150	0.54	63	(Edson sandy loam)	80-110	*0.66
15	Benridge-Konocti loam	0-30	0.17	64	(Edson sandy loam)	-122	*0.49
16	Benridge-Konocti loam	30-60	0.23	65	(Harris sandy loam)	0-11	0.31
17	Benridge-Konocti loam	60-90	0.23	66	(Harris sandy loam)	11-36	0.42
18	Benridge-Konocti loam	90-120	0.30	67	(Jaystreet sandy loam)	0-17	0.59
19	Benridge-Konocti loam	120-150	0.30	68	(Jaystreet sandy loam)	17-37	0.35
20	Benridge-Konocti loam	0-30	0.09	69	(Macintosh sandy loam)	0-13	0.38
21	Benridge-Konocti loam	30-60	0.29	70	(Macintosh sandy loam)	13-34	0.52
22	Benridge-Konocti loam	60-90	0.30	71	(McGavic gravelly sandy loam)	0-10	0.31
23	Benridge-Konocti loam	90-120	0.27	72	(McGavic gravelly sandy loam)	10-30	0.63
24	Benridge-Konocti loam	120-150	0.26	73	(McGavic gravelly sandy loam)	30-47	0.91
25	Glenview-Arrowhead loam	0-30	-0.09	74	Mounthat loam	0-30	0.31
26	Glenview-Arrowhead loam	30-60	0.07	75	Mounthat loam	0-30	0.31
27	Glenview-Arrowhead loam	60-90	0.19	76	Mounthat loam	30-60	0.35
28	Glenview-Arrowhead loam	90-120	0.18	77	Mounthat loam	60-90	0.59
29	Glenview-Arrowhead loam	120-150	0.19	78	Mounthat loam	90-120	*0.80
30	Vitrando-Cinderland loam	0-30	0.38	79	Mounthat loam	120-150	*1.08
31	Vitrando-Cinderland loam	30-60	0.00	80	Mounthat loam	150-180	*1.40
32	Vitrando-Cinderland loam	0-30	0.03	81	(Mudwell sandy loam)	0-15	-1.75
33	Whispering-Collayomi loam	0-30	0.21	82	(Mudwell sandy loam)	15-38	-1.50
34	Whispering-Collayomi loam	30-60	0.21	81	(Mudwell sandy loam)	0-15	0.36
35	Whispering-Collayomi loam	30-60	0.38	82	(Mudwell sandy loam)	15-38	0.57
SHASTA COUNTY VOLCANIC SOILS				83	Ponto sandy loam	0-30	0.24
36	Unnamed soil 1693-4	—	0.14	84	Ponto sandy loam	30-60	0.24
37	Unnamed soil 1693-5	—	0.14	85	Ponto sandy loam	60-90	0.24
38	(Cabin Butte sandy loam)	0-13	0.14	86	Ponto sandy loam	90-120	0.10
39	(Cabin Butte sandy loam)	13-34	0.21	87	Ponto sandy loam	120-150	0.07
40	(Clear Creek loam)	0-10	0.07	88	(Redtank sandy loam)	0-15	0.52
41	(Clear Creek loam)	10-41	0.14	89	(Redtank sandy loam)	15-44	0.77
42	(Clear Creek loam)	41-53	0.38	90	(Redtank sandy loam)	44-63	0.59
43	(Clear Creek loam)	53-65	0.45	91	Wyntoon loam	0-20	-1.26
44	(Clear Creek loam)	65-71	0.45	92	Wyntoon loam	20-41	-0.80
45	(Clear Creek loam)	71-80	0.52	91	Wyntoon loam	0-20	0.32
46	(Clear Creek loam)	80-84	0.49	92	Wyntoon loam	20-41	0.36

() Unofficial soil name. Brief profile descriptions are available from the U.S. Forest Service, P.O. Box 1620, McCloud, CA 96057.

* Some iodide was oxidized to iodine. See text, page 8.

The mean iodide sorption by volcanic soils in the Clear Lake area of Lake County was 0.25 ± 0.14 meq I⁻/100 g moist soil. The mean for volcanic soils in the Shasta-Trinity National forest was 0.42 ± 0.18 meq I⁻/100 g moist soil. Iodide was sorbed by surface samples as well as subsurface samples, with no apparent pattern (Fig. 2).

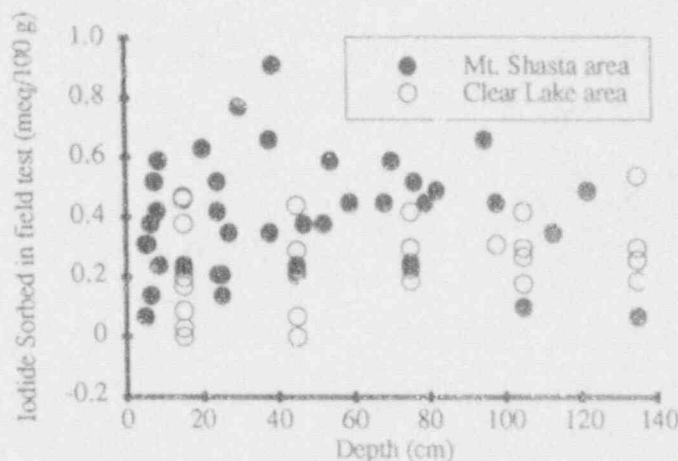


Fig. 2 Iodide sorption by volcanic soils of northern California

Four samples appeared to release iodide initially (shown as negative sorption in Table 1). When retested, their sorption of iodide was normal. All but one of the samples that sorbed < 0.1 meq I⁻/100 g soil were taken from < 60 cm depth. Iodide sorption by sample 73 was exceptionally high, 0.91 meq I⁻/100 g moist soil.

We were unable to accurately measure iodide sorption on the samples marked with asterisks in Table 1. These samples oxidized iodide in repeated tests, as was evident from bright yellow leachates and pink coloration of translucent capped bottles containing soil saturated with potassium iodide solution. Still, even with iodide being lost through oxidation, only three samples (78, 79, and 80) removed unusually large amounts of iodide.

Iodide sorption by air-dry samples 36 and 37 was 0.14 meq I⁻/100 g soil.

Laboratory determination of anion exchange capacity

Table 2 (p. 9) includes both results of preliminary experiments that were necessary for methods development (Sierra Nevada soils) and results of our first efforts in testing samples that we had collected from volcanic terrain (Lake County and Shasta County soils).

Of the Sierra Nevada soils Aiken loam and Challenge clay loam are non-volcanic soils that had been stored in an air-dry condition. Windy loam is a volcanic soil that had been kept continuously moist in storage. Our first laboratory determinations of anion exchange capacity (Table 2) showed 0.17 meq Cl⁻/100 g soil in Windy loam, where we hoped to find it, but none in Aiken loam or Challenge clay loam. These results encouraged us to continue development of the iodide sorption field test.

Anion retention in soil: development of a field test

TABLE 2
CHLORIDE-NITRATE EXCHANGE ON WHOLE SOILS FROM NORTHERN CALIFORNIA

Date of Experiment	Sample	Soil type	Depth	Chloride in extract	Chloride entrained	Mass of dry soil	Chloride exchanged
			cm	meq	meq	g	meq/100g
SIERRA NEVADA SOILS							
3/5/92	1	Aiken loam	0-30	0.017	0.014	14.2	0.00
3/5/92	1	Aiken loam	0-30	0.016	0.014	14.2	0.00
3/5/92	1	Aiken loam	0-30	0.019	0.014	14.2	0.00
3/5/92	1	Aiken loam	0-30	0.018	0.014	14.2	0.00
3/5/92	2	Challenge clay loam	0-30	0.010	0.014	14.8	0.00
3/5/92	2	Challenge clay loam	0-30	0.011	0.014	14.8	0.00
3/5/92	2	Challenge clay loam	0-30	0.011	0.014	14.8	0.00
3/5/92	2	Challenge clay loam	0-30	0.024	0.014	14.9	0.00
3/5/92	3	Windy loam (volcanic)	40-60	0.029	0.013	9.2	0.18
3/5/92	3	Windy loam (volcanic)	40-60	0.029	0.013	9.2	0.18
3/5/92	3	Windy loam (volcanic)	40-60	0.027	0.013	9.1	0.16
3/5/92	3	Windy loam (volcanic)	40-60	0.027	0.013	9.1	0.16
LAKE COUNTY VOLCANIC SOILS							
6/12/92	14	Benridge-Konocti clay loam	120-150	0.040	0.020	14.5	0.14
6/12/92	14	Benridge-Konocti clay loam	120-150	0.044	0.018	14.5	0.18
6/12/92	14	Benridge-Konocti clay loam	120-150	0.039	0.018	14.5	0.14
6/12/92	25	Glenview-Arrowhead loam	0-30	0.005	-0.003	15.5	-0.02
6/12/92	25	Glenview-Arrowhead loam	0-30	0.006	-0.003	15.5	-0.02
6/12/92	25	Glenview-Arrowhead loam	0-30	0.011	-0.003	15.5	-0.02
SHASTA COUNTY VOLCANIC SOILS							
8/6/92	59	Depner loam	120-150	0.028	0.007	10.2	0.21
8/6/92	59	Depner loam	120-150	0.021	0.007	10.2	0.14
8/6/92	63	(Edson sandy loam)	80-110	0.050	0.012	10.5	0.36
8/6/92	63	(Edson sandy loam)	80-110	0.026	0.008	10.5	0.17
8/6/92	73	(McGavic gravelly sandy loam)	30-47	0.092	0.010	10.9	0.74
8/6/92	73	(McGavic gravelly sandy loam)	30-47	0.078	0.011	10.9	0.61
8/6/92	80	Mounthat clay loam	150-180	0.039	0.011	9.5	0.30
8/6/92	80	Mounthat clay loam	150-180	0.027	0.009	9.5	0.18
8/20/92	36	Unnamed soil 1693-4	—	0.008	0.004	10.0	0.03
8/20/92	36	Unnamed soil 1693-4	—	0.009	0.004	10.0	0.04
8/20/92	37	Unnamed soil 1693-5	—	0.005	0.004	10.0	0.03
8/20/92	37	Unnamed soil 1693-5	—	0.002	0.004	10.0	0.04
8/20/92	73	(McGavic gravelly sandy loam)	30-47	0.116	0.011	11.0	0.97
8/20/92	73	(McGavic gravelly sandy loam)	30-47	0.109	0.011	10.9	0.90
8/20/92	89	(Redtank sandy loam)	15-44	0.018	0.007	10.8	0.10
8/20/92	89	(Redtank sandy loam)	15-44	0.019	0.007	10.8	0.11

() Unofficial soil name.

Chloride–nitrate exchange capacity of the samples from Lake County and Shasta County ranged from 0.00 to 0.94 meq Cl^- /100 g soil, with a mean of 0.31 ± 0.3 .

Anion exchange capacity of air-dry samples 36 and 37 was ≤ 0.04 meq Cl^- /100 g soil.

Validation of the iodide sorption field test

The crux of our work is validation of the iodide sorption field test as a semiquantitative measure of anion exchange capacity in natural soils. In deciding which samples to select for quantitative anion exchange capacity measurements, our selection criteria for the Lake County and Shasta County samples were low iodide sorption in the field test (sample 25), high iodide sorption (samples 14, 63, 73 and 89), and unmeasurable iodide sorption (samples 59 and 80) (See Tables 1 and 2, pp. 7 and 9).

Because anion sorption is a more general process than anion exchange, a strong direct relationship between iodide sorption and anion exchange capacity was not expected. Figure 3 shows a weak relationship between anion exchange capacity and iodide sorbed in the field test. This relationship was obtained by excluding McGavie (30–47 cm) from the regression.

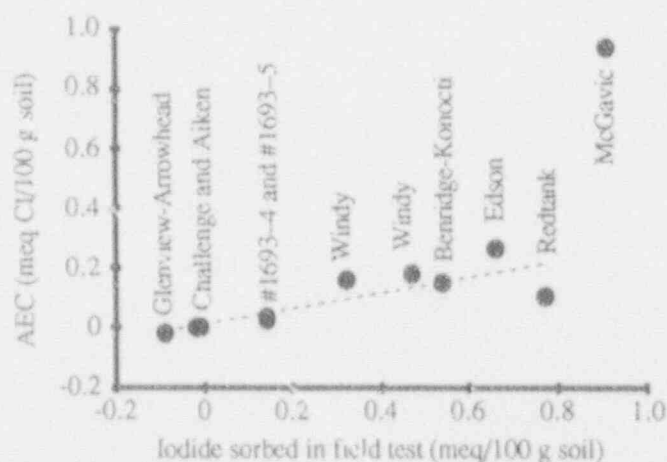


Fig. 3 Iodide sorbed in field test vs. nitrate–chloride exchange capacity.

We attempted to improve the relationship by measuring iodide sorption after shaking a fresh set of suspensions for 1 hour on a mechanical shaker (Fig. 4). The result was a weakening of the relationship. Samples that sorbed highly in the field test sorbed less after shaking, medial samples sorbed about the same, and low-sorbing samples sorbed more after shaking. Extended vigorous shaking resulted in a general levelling of iodide sorption.

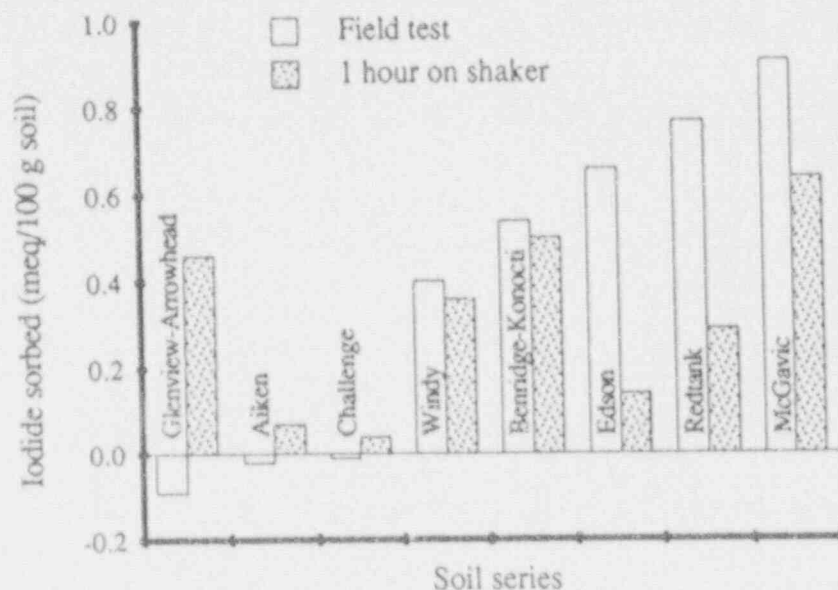


Fig. 4 Iodide sorbed during field test and after 1 hour of mechanical shaking at 190 cycles per minute.

DISCUSSION

Summary assessment of iodide field test data

Reviewing all our data, we note that:

- (i) All samples that sorbed iodide at levels ≥ 0.34 meq I⁻/100 g soil in the field test showed anion exchange capacity > 0.04 meq Cl⁻/100 g soil.
- (ii) The test showed no false negatives (cases where iodide sorption is zero and the sample shows anion exchange capacity).
- (iii) The test showed no false positives (cases where the test predicts anion exchange capacity that is not there).

Returning to STATEMENT OF THE PROBLEM, perhaps the most important result we found is

Maximum field test sorption = Maximum anion exchange capacity (McGavic).

Anion exchange capacity and anion retention

If volcanic soil were eventually to be used for conditioning the near-field environment around LLW disposal units to retard the migration of anionic solutes, an important mechanism would be anion retention analogous to (if not identical with) that which operates

in chromatographic columns. As we are interested in the ability of soils to retain anions during solute transport through soil, it might be worthwhile to consider intermediate data obtained in the anion exchange experiment of August 20, 1992 (Table 3):

TABLE 3
INTERMEDIATE DATA FROM CHLORIDE-NITRATE EXCHANGE TEST

Sample number	Soil name	Mass of entrained Cl^- solution	Conductivity of last rinse	Cl^- conc. of last rinse	Chloride entrained
		g	$\mu\text{S}/\text{cm}$	meq/L	meq
73	McGavic	5.90	0.269	1.79	0.011
73	McGavic	5.66	0.296	1.98	0.010
89	Redtank	4.77	0.224	1.49	0.007
89	Redtank	5.01	0.199	1.31	0.007
36	Soil 1693-4	3.52	0.192	1.27	0.004
36	Soil 1693-4	3.61	0.188	1.24	0.004
37	Soil 1693-5	3.08	0.185	1.22	0.004
37	Soil 1693-5	3.25	0.203	1.34	0.004

Each sample was rinsed equally four times with 50 ml of 0.001 *N* KCl, yet McGavic retained 33% more entrained chloride than Redtank, the second ranked soil. The electrical conductivity of the final rinses of the McGavic samples was about 282 $\mu\text{S}/\text{cm}$ and that of the Redtank samples was about 211 $\mu\text{S}/\text{cm}$. Apparently, high anion exchange capacity in a soil can be effective in retaining anions against solute transport.

General factors affecting sorption and exchange

We know that change in pH can have important effects on anion exchange capacity in variable-charge soils. Soil structure, soil drying, and electrolyte concentration of soil solutions can also affect sorption and exchange.

a) Soil structure

Undisturbed soils are variegated and delicate natural bodies. Small mineral particles are bound together with both mineral and organic coatings or filaments, some of which are products of microorganisms living in the soil. We have carried out our experimental work on samples that we describe as 'whole soils'. That is, we have excavated and sifted the soil samples, but we have not deliberately tried to disrupt the aggregates that were formed in natural environments.

Mechanical disruption of soil structure may affect anion sorption in whole soils (See Fig. 4). Field-test sorption changed little between 1 and 18 hours after initiation when disruption of soil structure was minimized. But sorption extremes were smoothed out by extended shaking of samples that reduced structure present in whole soils. We intend that the material we test be similar to that which would be used to line a LLW facility.

b) Drying of soil

The effects of air-drying of soil samples are not always predictable. Air-drying of samples could possibly affect both soil structure and anion sorption. Birrell and Fieldes (1952) examined volcanic ash soils with high natural water-holding capacity that shrank and lost much of their water-holding capacity when air-dried. The Soil Survey Staff (1975) recommends that samples of Andepts (Andisols in the latest soil taxonomy) be kept moist, because air-drying might affect cation exchange capacity (CEC):

Andepts in a perhumid climate are never dry while they remain in place. They lose CEC if allowed to become air dry before the determination is made. The values used in [taxonomic] definitions are those determined on soil samples that have never been allowed to dry, but the values have been recalculated to an oven-dry basis. The CEC of samples that have been air dried may be used if it has been determined that drying does not affect the CEC of that soil (Soil Survey Staff 1975, p.233).

Gebhardt and Coleman (1974) used air-dried soils in their experiments after limited work with Mexican samples showed no appreciable effect of air drying on Cl adsorption.

We tested air-dried samples of Aiken loam and Challenge clay loam in developing our methods. We do not know the effects of air-drying these samples. Validation of the iodide sorption field test is based on results obtained with moist samples freshly collected from volcanic terrain.

S. B. Southard, University of California, Davis, provided two air dry samples (36 and 37, Tables 1 and 2). According to Southard's measurements they contained 17% allophane/imogolite by weight. These samples ranked very low in both iodide sorption and anion exchange capacity. We do not know if these low results are a consequence of air drying the samples or are due to other factors.

c) Ionic strength

Table 4 (p. 14) is a compilation of data from several authors showing the magnitude and variability of anion exchange capacity in natural allophanic clays separated from volcanic soils. Investigations of the anion exchange capacity of allophanic clays have often been done by clay mineralogists who have tested the $< 2 \mu\text{m}$ fraction of soil clays separated from soils, and for this reason alone the data is not directly comparable to our data for whole soils.

But also, because 'ion exchange capacity' increases with increasing electrolyte concentration of equilibrating solutions (Barrow 1987) data obtained at different electrolyte concentrations cannot be compared directly. Calculations of anion exchange capacity can be precise when the amount of entrained chloride left in samples before extraction with nitrate is small (See Table 3). With a view to preventing hydrolysis (displacement of Cl^- by OH^- on exchange sites) other investigators have used electrolyte concentrations of equilibrating

solutions 30 to 60 times greater than ours. The rationale for choosing equilibrating solutions of 0.05 N KCl was documented by Fey and Le Roux (1976) :

A salt concentration of 0.05 *N* was found by Wada and Harada (1969) to be the optimum at which the opposing effects of hydrolysis in more dilute solutions, and non-Coulombic adsorption at higher concentrations, were smallest. Potassium was used as the index cation in this study because the small amount of cation-associated water in K-clays minimizes error attached to the inclusion of nonsolvent water into the weight basis for ion exchange capacity calculations. Chloride was chosen as index anion because of the minimal specificity with which it is adsorbed (Gebhardt and Coleman 1974, p. 255).

We feel that the Cl^-/OH^- ratio of 150,000 in 1.5×10^{-3} N KCl at pH 6 should make hydrolysis insignificant, while the low chloride concentration minimizes error in calculating the amount of entrained chloride. But a direct consequence of minimizing the electrolyte concentration of our equilibrating solutions is that our figures for anion exchange capacity will be lower than those of investigators who may use higher electrolyte concentrations.

TABLE 4
COMPENDIUM OF CHLORIDE-NITRATE EXCHANGE CAPACITY
DATA FOR ALLOPHANIC SOIL CLAYS AT pH 6

Reference	Ion Exchange Material	Soil Source	AEC pH 6	Equilibrating Solution	Solution Concentration
			<i>meq/100 g soil</i>		<i>N</i>
Clark and McBride, 1984	< 2 μ m clay	Egmont	12	NaCl	0.1
		KnP	2		
		Te Akatea	12		
Gonzales-Batista et al., 1982	< 2 μ m clay	Aguamansa-I	6.9	KCl	0.05
		Aguamansa-II	10.5		
		Akaka	6.2		
		Chio	3		
		Dos Cupressos	11		
		Las Aves	14.5		
Perrott, 1977	< 2 μ m clay	Te Akatea	6.5	NaCl	0.1
		Waihi	6.5		
Theng et al., 1982	< 2 μ m clay	Andesitic tephra	1.5	NaCl	0.05
		Imogolite	18		
		Rhyolitic tephra	5		
		Silica Springs	3		

Summary

Our goal is to locate soils with high natural anion exchange capacity that could possibly be used to retain anions in the near field environment of LLW facilities. In order to locate soil with sufficiently strong anion retention properties, we felt that a rapid semiquantitative field test for anion exchange capacity would be absolutely necessary. The test that we developed is based on the rapid sorption of iodide by soil samples.

Based on our experience with the iodide sorption field test, we suggest these tentative guidelines for identifying probable anion exchange capacity in soil samples:

Iodide sorbed (meq/100 g soil)	Probable anion exchange capacity
< 0.1	Negligible
0.1-0.5	Low
0.5-1.0	Moderate
>1.0	High

In our evaluation of the iodide sorption field test we found that:

- (i) All samples that sorbed iodide at levels ≥ 0.34 meq I⁻/100 g soil in the field test showed anion exchange capacity > 0.04 meq Cl⁻/100 g soil.
- (ii) The test showed no false negatives (cases where iodide sorption is zero and the sample shows anion exchange capacity).
- (iii) The test showed no false positives (cases where the test predicts anion exchange capacity that is not there).

Additionally, we found that the sample with the highest iodide sorption also had the highest anion exchange capacity.

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APPENDIX 1

SAMPLING COORDINATES FOR VOLCANIC SOILS FROM NORTHERN CALIFORNIA

Sample number and soil type	Latitude North	Longitude West	Sample number and soil type	Latitude North	Longitude West
SIERRA NEVADA SOILS			47 (Coonrod sandy loam)	41° 20' 16.4"	121° 59' 00.5"
1 Aiken loam	—	—	48 (Coonrod sandy loam)	41° 20' 16.4"	121° 59' 00.5"
2 Challenge clay loam	—	—	49 (Coonrod sandy loam)	41° 20' 16.4"	121° 59' 00.5"
3 Windy loam	—	—	50 (Coonrod sandy loam)	41° 20' 16.4"	121° 59' 00.5"
3 Windy loam	—	—	51 (Coonrod sandy loam)	41° 20' 16.4"	121° 59' 00.5"
LAKE COUNTY VOLCANIC SOILS			52 (Coonrod sandy loam)	41° 20' 16.4"	121° 59' 00.5"
4 Benridge-Konocti loam	39° 00' 47"	122° 40' 10"	53 (Coonrod sandy loam)	41° 20' 16.4"	121° 59' 00.5"
5 Benridge-Konocti loam	39° 00' 47"	122° 40' 10"	54 Depner sandy loam	40° 54' 27"	121° 45' 30"
6 Benridge-Konocti loam	39° 00' 47"	122° 40' 10"	55 Depner sandy loam	40° 54' 27"	121° 45' 30"
7 Benridge-Konocti loam	39° 00' 47"	122° 40' 10"	56 Depner sandy loam	40° 54' 27"	121° 45' 30"
8 Benridge-Konocti loam	38° 49' 45"	122° 32' 00"	57 Depner sandy loam	40° 54' 27"	121° 45' 30"
9 Benridge-Konocti loam	38° 49' 43"	122° 31' 46"	58 Depner sandy loam	40° 54' 27"	121° 45' 30"
10 Benridge-Konocti loam	38° 55' 13"	122° 39' 21"	59 Depner sandy loam	40° 54' 27"	121° 45' 30"
11 Benridge-Konocti loam	38° 55' 13"	122° 39' 21"	60 (Edson sandy loam)	41° 23' 27.5"	121° 47' 38.4"
12 Benridge-Konocti loam	38° 55' 13"	122° 39' 21"	61 (Edson sandy loam)	41° 23' 27.5"	121° 47' 38.4"
13 Benridge-Konocti loam	38° 55' 13"	122° 39' 21"	62 (Edson sandy loam)	41° 23' 27.5"	121° 47' 38.4"
14 Benridge-Konocti loam	38° 55' 13"	122° 39' 21"	63 (Edson sandy loam)	41° 23' 27.5"	121° 47' 38.4"
15 Benridge-Konocti loam	38° 55' 29"	122° 39' 08"	64 (Edson sandy loam)	41° 23' 27.5"	121° 47' 38.4"
16 Benridge-Konocti loam	38° 55' 29"	122° 39' 08"	65 (Harris sandy loam)	41° 24' 51.6"	121° 47' 10.0"
17 Benridge-Konocti loam	38° 55' 29"	122° 39' 08"	66 (Harris sandy loam)	41° 24' 51.6"	121° 47' 10.0"
18 Benridge-Konocti loam	38° 55' 29"	122° 39' 08"	67 (Jaystreet sandy loam)	41° 25' 22.1"	121° 47' 10.3"
19 Benridge-Konocti loam	38° 55' 29"	122° 39' 08"	68 (Jaystreet sandy loam)	41° 25' 22.1"	121° 47' 10.3"
20 Benridge-Konocti loam	40° 00' 57"	122° 38' 08"	69 (Macintosh sandy loam)	Shasta-Trinity Nat. Forest	
21 Benridge-Konocti loam	40° 00' 57"	122° 38' 08"	70 (Macintosh sandy loam)	Shasta-Trinity Nat. Forest	
22 Benridge-Konocti loam	40° 00' 57"	122° 38' 08"	71 (McGavic gr. sandy loam)	41° 26' 57.3"	121° 49' 13.2"
23 Benridge-Konocti loam	40° 00' 57"	122° 38' 08"	72 (McGavic gr. sandy loam)	41° 26' 57.3"	121° 49' 13.2"
24 Benridge-Konocti loam	40° 00' 57"	122° 38' 08"	73 (McGavic gr. sandy loam)	41° 26' 57.3"	121° 49' 13.2"
25 Glenview-Arrowhead loam	38° 54' 54"	122° 45' 06"	74 Mounthal loam	40° 53' 29.7"	121° 47' 50.1"
26 Glenview-Arrowhead loam	38° 54' 54"	122° 45' 06"	75 Mounthal loam	40° 53' 29.7"	121° 47' 50.1"
27 Glenview-Arrowhead loam	38° 54' 54"	122° 45' 06"	76 Mounthal loam	40° 53' 29.7"	121° 47' 50.1"
28 Glenview-Arrowhead loam	38° 54' 54"	122° 45' 06"	77 Mounthal loam	40° 53' 29.7"	121° 47' 50.1"
29 Glenview-Arrowhead loam	38° 54' 54"	122° 45' 06"	78 Mounthal loam	40° 53' 29.7"	121° 47' 50.1"
30 Vitrandepts-Cinderland loam	40° 00' 04"	122° 38' 07"	79 Mounthal loam	40° 53' 29.7"	121° 47' 50.1"
31 Vitrandepts-Cinderland loam	40° 00' 04"	122° 38' 07"	80 Mounthal loam	40° 53' 29.7"	121° 47' 50.1"
32 Vitrandepts-Cinderland loam	39° 00' 47"	122° 38' 10"	81 (Mudwell sandy loam)	41° 18' 28.6"	121° 45' 57.6"
33 Whispering-Collayomi loam	38° 49' 13"	122° 43' 30"	82 (Mudwell sandy loam)	41° 18' 28.6"	121° 45' 57.6"
34 Whispering-Collayomi loam	38° 49' 13"	122° 43' 30"	81 (Mudwell sandy loam)	41° 18' 28.6"	121° 45' 57.6"
35 Whispering-Collayomi loam	38° 49' 13"	122° 43' 30"	82 (Mudwell sandy loam)	41° 18' 28.6"	121° 45' 57.6"
SHASTA COUNTY VOLCANIC SOILS			83 Ponto sandy loam	41° 15' 46.4"	122° 10' 17.8"
36 Unnamed soil 1693-4	—	—	84 Ponto sandy loam	41° 15' 46.4"	122° 10' 17.8"
37 Unnamed soil 1693-5	—	—	85 Ponto sandy loam	41° 15' 46.4"	122° 10' 17.8"
38 (Cabin Butte sandy loam)	41° 20' 59.7"	121° 47' 13.0"	86 Ponto sandy loam	41° 15' 46.4"	122° 10' 17.8"
39 (Cabin Butte sandy loam)	41° 20' 59.7"	121° 47' 13.0"	87 Ponto sandy loam	41° 15' 46.4"	122° 10' 17.8"
40 (Clear Creek loam)	41° 20' 42.3"	122° 03' 25.2"	88 (Redtank sandy loam)	41° 18' 15.3"	121° 46' 23.9"
41 (Clear Creek loam)	41° 20' 42.3"	122° 03' 25.2"	89 (Redtank sandy loam)	41° 18' 15.3"	121° 46' 23.9"
42 (Clear Creek loam)	41° 20' 42.3"	122° 03' 25.2"	90 (Redtank sandy loam)	41° 18' 15.3"	121° 46' 23.9"
43 (Clear Creek loam)	41° 20' 42.3"	122° 03' 25.2"	91 Wyntoon loam	41° 20' 54.2"	121° 45' 30.6"
44 (Clear Creek loam)	41° 20' 42.3"	122° 03' 25.2"	92 Wyntoon loam	41° 20' 54.2"	121° 45' 30.6"
45 (Clear Creek loam)	41° 20' 42.3"	122° 03' 25.2"	91 Wyntoon loam	41° 20' 54.2"	121° 45' 30.6"
46 (Clear Creek loam)	41° 20' 42.3"	122° 03' 25.2"	92 Wyntoon loam	41° 20' 54.2"	121° 45' 30.6"

() Unofficial soil name. Brief profile descriptions are available from the U.S. Forest Service, P.O. Box 1620, McCloud, CA 96057.

APPENDIX 2

Measurement of chloride in 1 *N* potassium nitrate using a Cotlove chloride titrator.

A 1 meq Cl^-/L standard was prepared in 1 *N* potassium nitrate. In order to test the accuracy of chloride titration measurements in this matrix, serial dilutions of the standard were measured with the Cotlove chloride titrator.

The test showed that concentrations of chloride less than 0.7 meq Cl^-/L were not effectively measured. The problem was corrected by adding 0.7 meq Cl^-/L (as sodium chloride) to the polyvinyl alcohol-acetic acid mixture used in the titration. Careful 1 ml additions of the mixture to titration vials supplied a base level of chloride upon which chloride introduced with samples was measured.

Subsequent measurements of the serially diluted standards showed that chloride was accurately measured (Fig. 5).

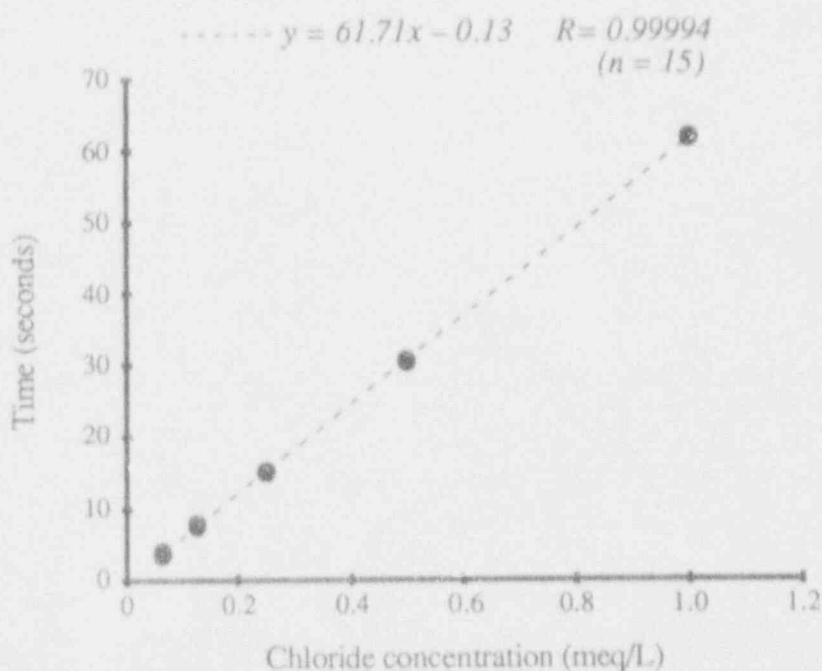
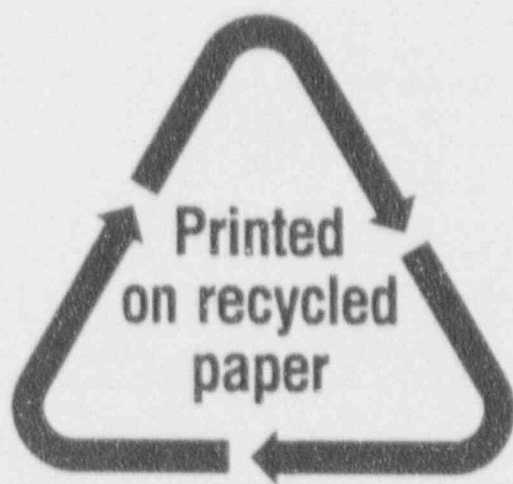


Fig. 5 The time required to titrate chloride standards in a matrix of 1 *N* potassium nitrate is proportional to the chloride concentration of the standards if measured on top of a base-level concentration of 0.7 meq Cl^-/L .

NRC FORM 335 (2-89) NRCM 1102, 3201, 3202	U.S. NUCLEAR REGULATORY COMMISSION BIBLIOGRAPHIC DATA SHEET <i>(See instructions on the reverse)</i>	1. REPORT NUMBER (Assigned by NRC. Add vol., Supp., Rev., and Addendum Numbers, if any.) NUREG/CR-5974									
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ANION RETENTION IN SOIL: POSSIBLE APPLICATION TO REDUCE
MIGRATION OF IODINE-131 AND IODINE-129

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