

EVALUATION OF ISOTOPE MIGRATION - LAND BURIAL

WATER CHEMISTRY AT COMMERCIALY OPERATED LOW-LEVEL RADIOACTIVE WASTE DISPOSAL SITES

Quarterly Progress Report
October - December 1981

Richard F. Pietrzak and Ramesh Dayal

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ABSTRACT

In this report we present field measurements of trench water properties conducted during the October 1981 sampling trip to Maxey Flats. In-line measurements of specific conductance, dissolved oxygen, Eh, pH, sulfide electrode response and temperature are reported. Appreciable changes in the specific conductance, relative to past results, were observed but since water is periodically removed and replenished by rainwater infiltration, fluctuations in the ionic strength of the solution are expected. Dissolved oxygen levels and pH remained relatively constant with respect to previous measurements. The oxidation potential, Eh, continues to evolve towards a more reducing condition. Sulfide concentrations were too low to be quantified.

Radionuclide sorption isotherms were evaluated for two Barnwell trench waters and sediment from the Hawthorne formation. The sorption data for ^{241}Am , ^{85}Sr , and $^{134,137}\text{Cs}$ could be represented by the linear isotherm which defines K_d . Sorption data for ^{60}Co could not be described completely by either the linear or Freundlich isotherm. Only a minimum value for the sorption coefficient, K_d , could be calculated. Comparison of ^{60}Co K_d data, based on batch sorption test using actual trench water and column loop test using synthetic water, indicates that in-situ complexing agents may be responsible for the lower ^{60}Co sorption results obtained by the isotherm method.

Laboratory experiments to study the chemical changes and the coprecipitation of radionuclides by ferric hydroxide formed during the oxidation of Maxey Flats and West Valley trench waters were made. During the oxidation of a trench 27 water from Maxey Flats, approximately 80% of the iron and all of the ^{137}Cs , ^{85}Sr , and ^{60}Co remained in solution. In similar experiments with a West Valley trench 8 water, essentially all of the iron precipitated but approximately 80% of the ^{60}Co and all of the ^{137}Cs and ^{85}Sr remained in solution.

An integrated waste form leaching test is being initiated to approximate the leaching of solidified waste and radionuclide transport through sediment in a partially water saturated environment. The waste form, consisting of IRN-77 ion exchange resin loaded with ^{85}Sr and ^{137}Cs and solidified with Portland I cement, will be surrounded with sediment from the Hawthorne formation at the Barnwell site. The radionuclide activity of the eluate will be measured periodically for a period of two months. At the conclusion of each experiment, the residual activity in the waste form and sediment will be determined. Release rates of radionuclides from the waste form will be evaluated.

CONTENTS

ABSTRACT	iii
CONTENTS	v
FIGURES.	vi
TABLES	vii
ACKNOWLEDGMENTS.	ix
1. WATER SAMPLE COLLECTION AT THE MAXEY FLATS DISPOSAL SITE, OCTOBER 1981	1
1.1 Introduction.	1
1.2 Experimental.	1
1.3 Results and Discussion.	1
2. RADIONUCLIDE SORPTION ISOTHERMS FOR MATERIALS FROM THE BARNWELL DISPOSAL SITE.	5
2.1 Introduction.	5
2.2 Experimental.	6
2.3 Results and Discussion.	8
3. CHEMICAL CHANGES AND RADIONUCLIDE SCAVENGING DURING THE OXIDATION OF TRENCH WATER.	19
3.1 Introduction.	19
3.2 Materials	19
3.3 Experimental Procedures	20
3.4 Results and Discussion.	21
4. AN INTEGRATED WASTE FORM/SEDIMENT LEACHING TEST.	29
4.1 Introduction.	29
4.2 Materials	29
4.2.1 Waste Form	29
4.2.2 Leachant	30
4.2.3 Sediment	30
4.3 Apparatus	30
4.4 Leaching Procedure.	32
4.5 Data Analyses	32
5. REFERENCES	33

FIGURES

1.1	Locator Map of the Maxey Flats Disposal Site Showing the Location and Identification of the Disposal Trenches.	2
2.1	²⁴¹ Am Sorption From Barnwell Trench Sump 6D1 Water on Hawthorne Sediment.	10
2.2	²⁴¹ Am Sorption From Barnwell Trench Sump 25-21-D1 Water on Hawthorne Sediment.	10
2.3	⁸⁵ Sr Sorption From Barnwell Trench Sump 6D1 Water on Hawthorne Sediment.	11
2.4	¹³⁴ Cs Sorption From Barnwell Trench Sump 6D1 Water on Hawthorne Sediment.	12
2.5	¹³⁴ Cs Sorption From Barnwell Trench Sump 25-21-D1 Water on Hawthorne Sediment.	12
2.6	¹³⁷ Cs Sorption From Barnwell Trench Sump 6D1 Water on Hawthorne Sediment.	13
2.7	¹³⁷ Cs Sorption From Barnwell Trench Sump 25-21-D1 Water on Hawthorne Sediment.	13
2.8	⁶⁰ Co Sorption From Barnwell Trench Sump 6D1 Water on Hawthorne Sediment.	14
2.9	⁶⁰ Co Sorption From Barnwell Trench Sump 25-21-D1 Water on Hawthorne Sediment.	14
3.1	Changes in the pH, Eh, and Metal Concentrations as a Function of Time During the Oxidation of Trench 27 Water From the Maxey Flats Disposal Site	22
3.2	Changes in the pH, Eh, and Metal Concentrations as a Function of Time During the Oxidation of Trench 8 Water From the West Valley Disposal Site	23
3.3	Changes in the pH, Eh, and Metal Concentrations as a Function of Time During the Oxidation of Trench 8 Water From the West Valley Disposal Site With Added 1×10^{-5} M Cs ⁺ and Sr ²⁺	23
3.4	Dissolved Iron Concentrations as a Function of Eh During the Oxidation of Trench Waters From the Maxey Flats and the West Valley Disposal Sites.	27
4.1	Apparatus for Continuous Capillary Water Flow Leaching of Solidified Waste Forms.	31

TABLES

1.1	Field Measurements of Water Samples Collected at the Maxey Flats Disposal Site.	3
2.1	Chemical Compositions of Trench Waters From the Barnwell Disposal Site.	7
2.2	Characteristics of the Hawthorne Sediment From the Barnwell Disposal Site.	8
2.3	Linear Regression Analysis of Radionuclide Sorption Data for Barnwell Trench Sump 6D1 Water and Hawthorne Sediment.	15
2.4	Linear Regression Analysis of Radionuclide Sorption Data for Barnwell Trench Sump 25/21-D1 Water and Hawthorne Sediment	15
2.5	Comparison of Batch, Column Loop and Isotherm K_d for Barnwell Trench Sump 6D1 Water and Hawthorne Sediment.	16
3.1	Energies of the Gamma Photons Used for Gamma-Ray Analysis of the Radionuclides	20
3.2	Changes in Alkalinity, Cation Concentrations, and Redox Potential Measured Before and After Oxidation of WV8 and MF27 Trench Waters.	22
3.3	Equilibrium Yield for Various Metals in the Filtrate and Precipitate From Oxidized Maxey Flats Trench 27 Water.	24
3.4	Analyses of the Filtrate After Removal of the Ferric Oxyhydroxide Precipitate From Maxey Flats Trench 27 Water.	25
3.5	Equilibrium Yield for Various Metals in the Filtrate and Precipitate From Oxidized West Valley Trench 8 Water	26
4.1	Composition of Synthetic Barnwell Trench Water	30

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1. WATER SAMPLE COLLECTION AT THE MAXEY FLATS DISPOSAL SITE, OCTOBER 1981
(J. Clinton, W. W. Becker, and R. F. Pietrzak)

1.1 Introduction

The chemical and radionuclide composition of trench waters is important in understanding radionuclide transport at shallow land burial sites. In the past, the Maxey Flats site has been sampled by BNL four times over a six-year period. A construction project has been initiated at the Maxey Flats burial site by the State of Kentucky to minimize rainwater infiltration by covering the disposal trenches with plastic sheeting. A sampling trip was undertaken to obtain water samples before the trenches were completely covered. In this report, we present the results of field measurements made on samples collected during the fifth sampling trip.

1.2 Experimental

Trench waters were collected by anaerobic procedures developed earlier (Weiss and Colombo, 1980). The trenches sampled were those that are reported to contain chelating agents (Cleveland and Rees, 1981). In-line measurements of temperature, specific conductance, dissolved oxygen, sulfide, Eh and pH were performed. Measurement of sulfide concentrations was made using a silver/silver sulfide in-line electrode system. A map of the Maxey Flats disposal site, showing the location of the various trenches is given in Figure 1.1.

1.3 Results and Discussion

Results of field measurements are presented in Table 1.1. Appreciable changes in the specific conductance of the water in trench sumps 33-L-8, 27E, and 7-3 were observed relative to previous measurements. Since water was removed periodically from the trenches at this site and rainwater infiltrates the trench cap replenishing what was removed, fluctuations in the ionic strength are expected. The oxygen levels in solution remained low and the pH did not change substantially. The Eh of solution is lower for this sampling than all previous data indicating that the trench waters are evolving towards more reducing conditions.

The sulfide electrode measurements were outside the calibration range for the quantitative determination of sulfide (Berner, 1963). Therefore, sulfide concentrations in the trench waters could not be calculated from the field measurements. Nevertheless, very low concentrations of sulfide ion could be present in the waters of trench sumps 19W, 27-E and 35. Sulfate

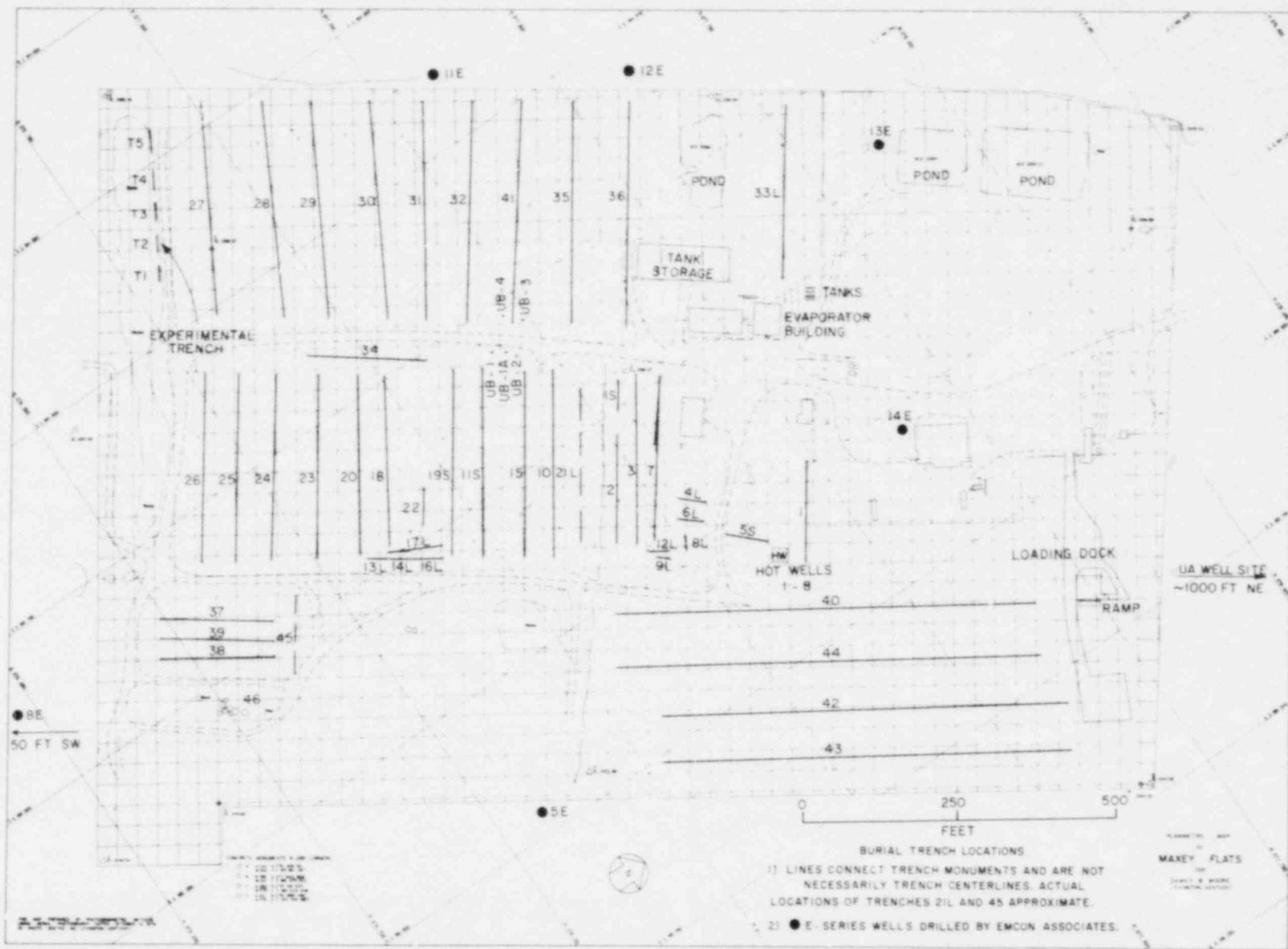


Figure 1.1 Map of the Maxey Flats disposal site, showing the location and identification of the disposal trenches. The exact location of the sump standpipes in each trench was not available.

reducing bacteria have been identified in the trench waters of the Maxey Flats site (Weiss and Colombo, 1980). The presence of sulfide ion could be an indication that sulfate reduction was taking place. In addition, preliminary data indicate that sulfate concentration in these trenches (19, 27, 35) were low (<1.5 mg/L). Nevertheless, most metal sulfides exhibit low solubility, resulting in the loss of sulfide from solution by in situ precipitation.

Table 1.1

Field Measurements of Water Samples Collected at the Maxey Flats Disposal Site

Sampling Location Trench	Date	Temperature (°C)	Specific Conductance (µmho/cm) ^a	Dissolved Oxygen (mg/L)	Eh (mV, NHE) ^b	pH	Sulfide (mV) ^c
33-L-8	10/81	18.0	2000	0.25	-135.4	6.0	-125
33-L-8	6/77	19.5	1060	d	d	6.6	e
33-L-4	10/81	17.0	6400	0.05	-53.9	12.0	-278
33-L-4	5/78 ^g	12.0	5580	4.1	-7.0	12.0	e
33-L-4	9/76 ^f	20.0	7600	d	d	12.3	e
23M	10/81	17.0	4800	0.05	-38.5	7.5	-302
23	6/77	23.0	g	d	d	7.2	e
27E	10/81	16.0	6600	<0.05	16.8	6.8	-353
27	10/79	18.0	18000	0.15	140.0	5.9	-490
27	5/78	17.6	9370	0.05	17.0	6.6	e
27	9/76 ^f	20.0	12000	d	d	6.0	e
35	10/81	17.0	3400	0.1	-13.9	8.2	-486
35	6/77	16.0	5500	d	d	7.9	e
19W	10/81	15.0	2100	0.1	-27.5	6.5	-465
19S	5/78	17.4	2310	0.2	25.0	6.9	e
19S	9/76 ^f	21.0	2340	d	d	6.6	e
7-3	10/81	16.5	12000	0.1	-43.6	7.4	-324
7	6/77	28.0	2330	d	d	7.1	e
7	9/76 ^f	27.5	2530	d	d	6.9	e

^aSpecific conductance expressed as µmho/cm at 25°C.

^bField measurements of Eh are reported relative to the normal hydrogen electrode (NHE).

^cThe field sulfide electrodes measurements are below the operating concentration range of the silver/silver sulfide electrode system.

^dRedox potential (Eh) and dissolved oxygen were not measured in September 1976.

^eSulfide was not measured until 1979.

^fIn September 1976, field measurements were made on separate aliquots of trench water within 30 minutes of collection. On subsequent trips, measurements were made with probes inserted directly into the flow stream.

^gField measurements not made.

2. RADIONUCLIDE SORPTION ISOTHERMS FOR MATERIALS FROM THE BARNWELL DISPOSAL SITE (R. Pietrzak)

2.1 Introduction

Mathematical models used to calculate radionuclide transport by groundwater generally assume ideal chromatographic behavior for radionuclide sorption. The distribution coefficient, K_d , must be experimentally determined to account for the partitioning of each radionuclide between the solution and the solid phases. The distribution coefficient, K_d , is defined as the ratio of equilibrium radionuclide concentration on the solid phase to the equilibrium concentration of radionuclide in solution. Many parameters and mechanisms control the concentration of radionuclides in solution: pH, ionic strength, complex formation, precipitation, etc. Changes in the radionuclide concentrations or in the chemistry of the solution will affect the observed values for K_d .

The equilibrium sorption isotherm is a mathematical relationship which describes the radionuclide distribution between the liquid and solid phase. The Freundlich isotherm is the simplest relationship used to define nonlinear behavior,

$$S = K C^N \quad (1)$$

or

$$\log S = \log K + N \log C \quad (2)$$

where K and N are constants.

and at equilibrium

S = concentration of solute sorbed on the sediment

C = concentration of solute in solution.

The Freundlich isotherm may be derived from Langmuir's theory of monolayer adsorption (Travis and Etnier, 1981). The simplest sorption isotherm is the linear relationship which defines K_d . If $N = 1$ in the Freundlich isotherm equation given in (1), then

$$S = K C \quad (3)$$

where

K = equilibrium distribution coefficient (K_d).

Graphical methods of presentation are used to see if radionuclide sorption data will fit an isotherm. The sensitivity of K_d to changes in experimental conditions and mechanisms which keep radionuclides in solution are

reflected in how well the experimental data fits an isotherm. The technique of graphical presentation of experimental sorption data to evaluate the fit to mathematical isotherm relationships may be useful for several reasons:

- The sorption behavior of the radionuclides over a range of experimental parameters can be easily seen.
- Abrupt changes in the sorption behavior can be interpreted as changes in the mechanisms which keep radionuclides in solution.
- Appropriate conditions and data for modeling calculations and further experimental work can be selected.

2.2 Experimental

Hydrochloric acid solutions of ^{60}Co , ^{85}Sr , ^{241}Am , and $^{137,134}\text{Cs}$ were evaporated to dryness. The radionuclide salts were dissolved in filtered Barnwell trench water. The isotope concentrations in the resultant solution were approximately $0.2 \mu\text{Ci/mL}$. The chemical compositions of the trench 6D1 and 25-21-D1 waters used in this study, are given in Table 2.1. The initial radionuclide concentrations of the experimental solutions were determined, in triplicate, on aliquots of spiked trench water.

The sediment used in this study was a silty sand from the Hawthorne Formation at the Barnwell site sampled by J. Cahill (U.S. Geological Survey) from a depth corresponding to the trench bottoms. The characteristics of the sediment are given in Table 2.2.

Table 2.1

Chemical Compositions of Trench Waters from
the Barnwell Disposal Site^a

	Concentration (mg/L) ^b	
	Trench 6D1	Trench 25-21-D1
Sodium	28	10.7
Potassium	3.0	1.2
Ammonia as N	3.7	4.1
Calcium	14	9.5
Magnesium	1.4	3.2
Iron	<1	6.1 ^c
Total CO ₂ ^d (as CaCO ₃)	70	370
Chloride	13.3	12.1
Sulfate	44.5	<5
Nitrate as N	4.2	<0.1
Silica	7.3	7.2
pH (unitless)	6.1	6.2
Eh (mV, NHE)	+350	+160
Specific Conductance (μmho/cm)	260	190

^aAs reported by Czyscinski and Weiss (1981).

^bExcept where units are specified.

^cFe²⁺/Fe³⁺ = 30.

^dProbe method [see Appendix A in Czyscinski and Weiss (1981)].

Table 2.2

Characteristics of the Hawthorne Sediment
from the Barnwell Disposal Site^a

Sand:silt:clay	75:10:15
Surface Area (m ² /g)	0.3-13
Cation Exchange Capacity (meq/100 g)	6.0
Organic carbon (wt %)	0.03
Extractable Iron (wt %)	0.2-0.3
Carbonate (wt %)	0.0
<u>Mineral Content</u>	
Quartz	85%
Mica	5-10%
Kaolinite	5-10%
Feldspar	<2%
Goethite	<5%
Hematite	<5%
Heavy Minerals	<2%
^a Weiss and Colombo (1980).	

Batch Sorption Method

Increasing amounts of sediment relative to a fixed volume of water (20-mL) were used so that the solution to soil ratio ranged from 20/1 to less than 5/1. Experimental controls consisted of aliquots of spiked trench water added to empty Teflon containers. The controls and samples were tumbled for three months to establish equilibrium. Periodically the samples were centrifuged to separate the water and sediment phases. The radionuclide activity in the water phase was measured on a Ge(Li) detector to determine when steady-state concentrations of the radionuclides in solution were achieved. When a steady state had been established, the samples were centrifuged and disassembled. The liquid phase of each sample or control was filtered through a 0.45 μ m membrane filter. The acidified aliquots were counted on a Ge(Li) detector to determine the activity of each radionuclide in solution.

2.3 Results and Discussions

The relative concentration of radionuclide sorbed on the solid phase was calculated from the relationship:

$$S = \frac{A_0 - A_E}{A_0} \cdot \frac{1}{W} \quad (4)$$

where

A_0 = initial radionuclide activity in solution;

A_E = final radionuclide activity in solution at equilibrium;

W = weight of the solid phase (g).

The fraction of radionuclide remaining in solution (C) was calculated as:

$$C = \frac{A_E}{A_0} \cdot \frac{1}{V} \quad (5)$$

where V = volume of solution (mL)

The experimental data were plotted on Cartesian and log-log coordinates to see if linear or nonlinear Freundlich sorption isotherms were followed. The Cartesian coordinate plots of the experimental data are shown in Figures 2.1 to 2.9, showing that the experimental data could be represented by linear sorption isotherms. Linear regression analyses of the sorption data are given in Tables 2.3 and 2.4.

The radionuclide sorption coefficients (K_d) reported earlier (Pietrzak et al., 1982) for trench 6D1 water and sediment from the Hawthorne formation at Barnwell are compared with the current results in Table 2.5. The results reported for batch K_d were obtained by tumbling a radionuclide spiked Barnwell trench 6D1 water with disaggregated Hawthorne sediment having a fixed solution-to-solid ratio (15:1). The column K_d results were obtained by recirculating a radionuclide spiked synthetic trench 6D1 water in a plug of intact sediment core from the Hawthorne formation. The results presented in this report are based on the sorption data obtained by using a batch sorption method described in Section 2.2. The solution-to-solid ratio ranged from 20:1 to 5:1.

^{241}Am showed a linear sorption behavior at low concentrations in solution (Figures 2.1 and 2.2). As shown in Tables 2.3 and 2.4, the ^{241}Am sorption coefficients are 1600 mL/g for 6D1 water and 86 mL/g for 25-21-D1 water. Sheppard et al. (1976) reported the distribution between soils from various locations and a solution containing ^{241}Am (III). They reported K_d values ranging from about 250 for Fuquay sand to 5000 mL/g for Muscatine silt loam. The results for Barnwell trench waters and sediment are comparable to K_d values reported by Sheppard. The ^{241}Am K_d values based on different techniques and using 6D1 water range from 1150 mL/g to 1600 mL/g (Table 2.5).

^{85}Sr was clearly represented by the linear sorption isotherm (Figure 2.3), giving a K_d value of 13 mL/g for 6D1 water (Table 2.3). Prout (1958, 1959) showed the strontium sorption on Savannah River soil to be pH dependent. The K_d values for ^{85}Sr , based on different methods and using 6D1 water, agree closely for the batch and isotherm method (Table 2.5). The sorption coefficient based on column loop method, however, is higher by more than a factor of two.

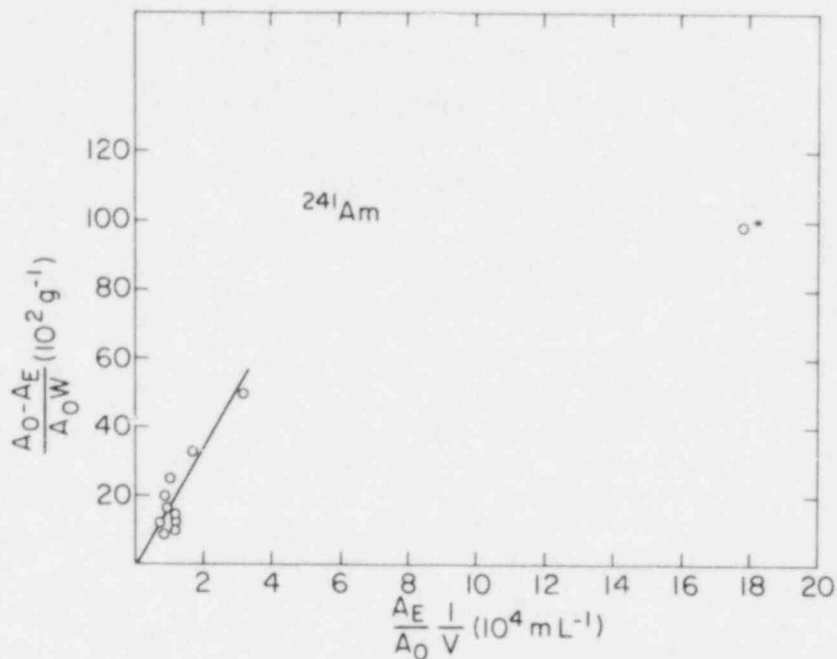


Figure 2.1 ^{241}Am sorption from Barnwell trench sump 6D1 water on Hawthorne sediment. The solid line represents the sorption isotherm, based on least squares linear regression. The data point indicated by * was not considered in the linear regression analysis.

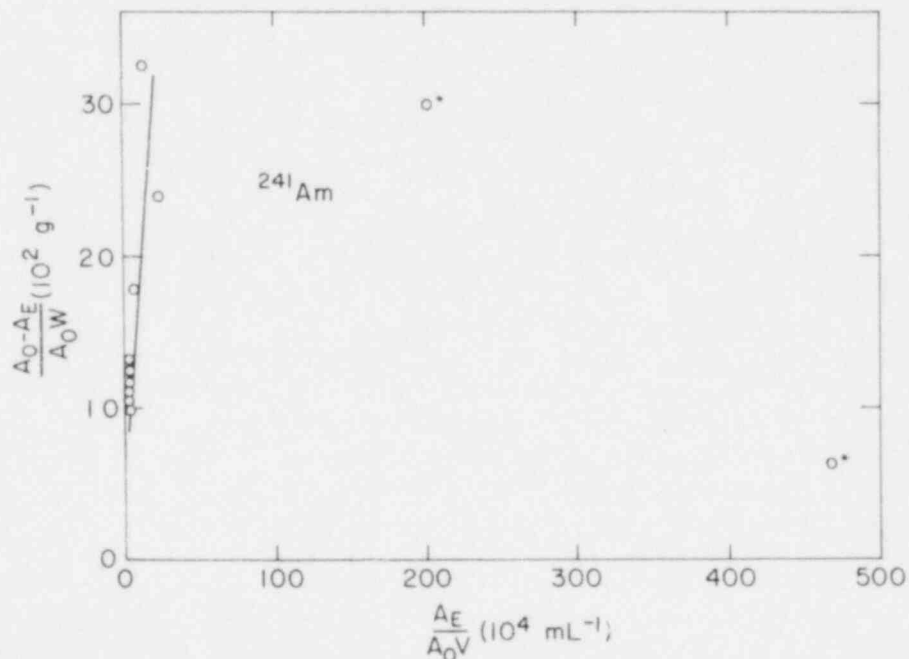


Figure 2.2 ^{241}Am sorption from Barnwell trench sump 25-21-D1 water on Hawthorne sediment. The solid line represents the least squares linear regression. The data points indicated by * were not considered in the linear regression.

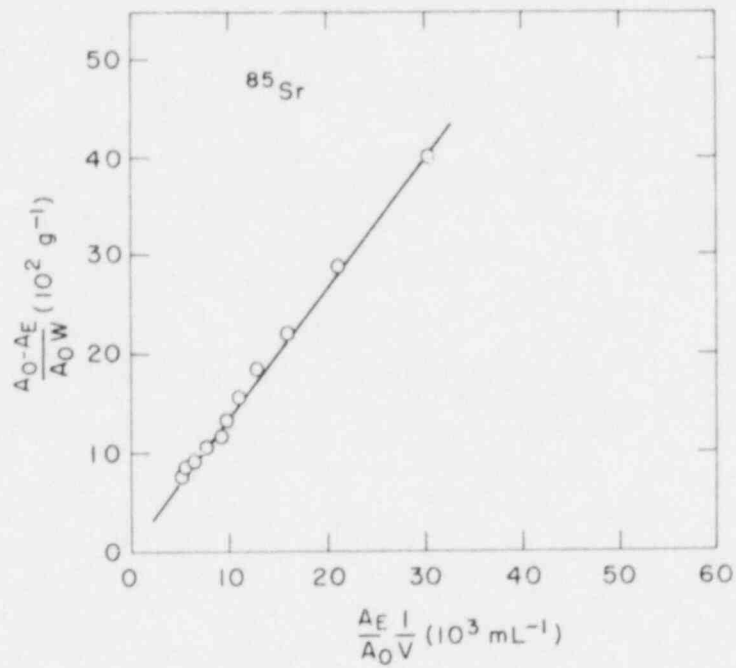


Figure 2.3 ^{85}Sr sorption from Barnwell trench sump 6D1 water on Hawthorne sediment. The solid line represents the least squares linear regression.

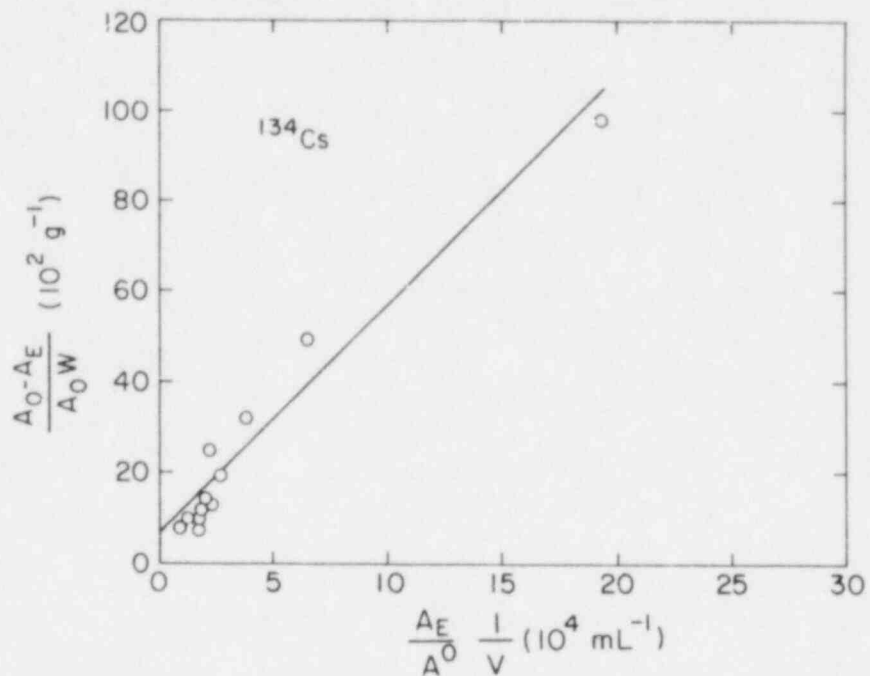


Figure 2.4 ^{134}Cs sorption from Barnwell trench sump 6D1 water on Hawthorne sediment. The solid line represents the least squares linear regression.

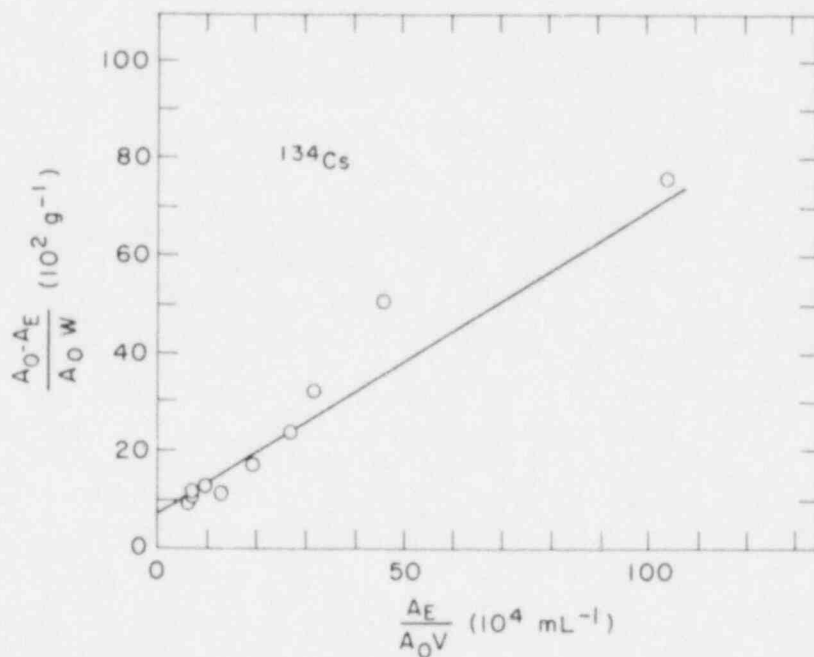


Figure 2.5 ^{134}Cs sorption from Barnwell trench sump 25-21-D1 water on Hawthorne sediment. The solid line represents the least squares linear regression.

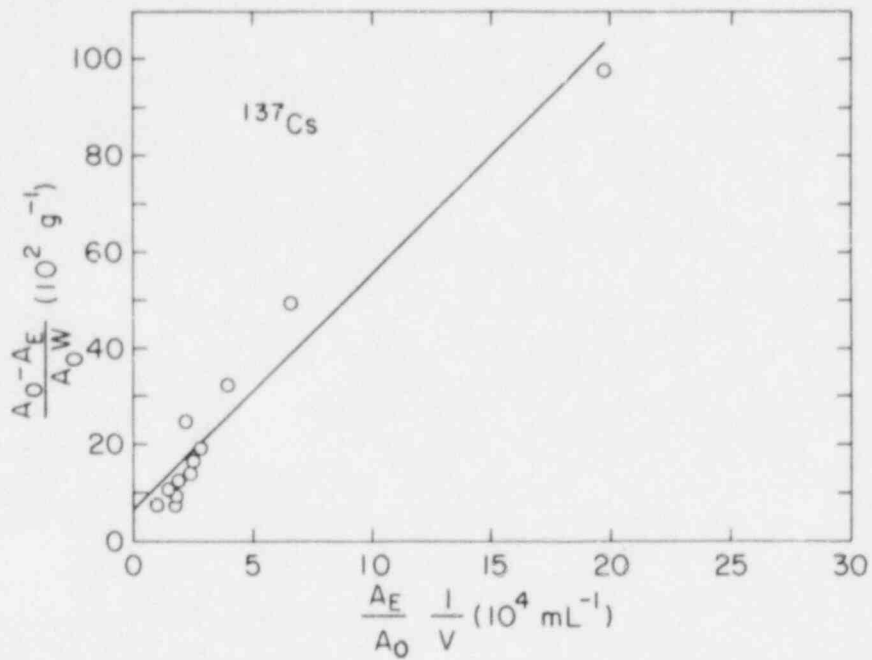


Figure 2.6. ^{137}Cs sorption from Barnwell trench sump 6D1 water on Hawthorne sediment. The solid line represents the least squares linear regression.

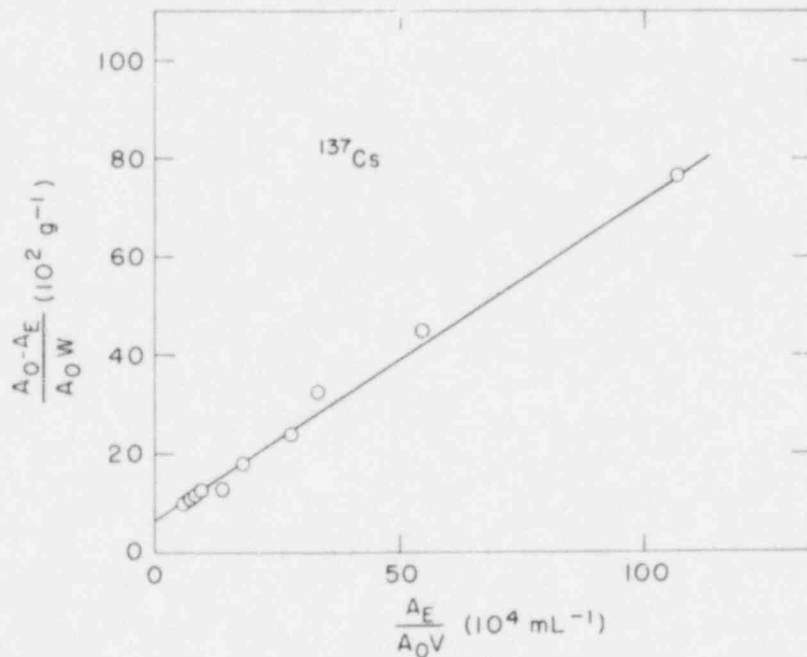


Figure 2.7. ^{137}Cs sorption from Barnwell trench sump 25-21-D1 water on Hawthorne sediment. The solid line represents the least squares linear regression.

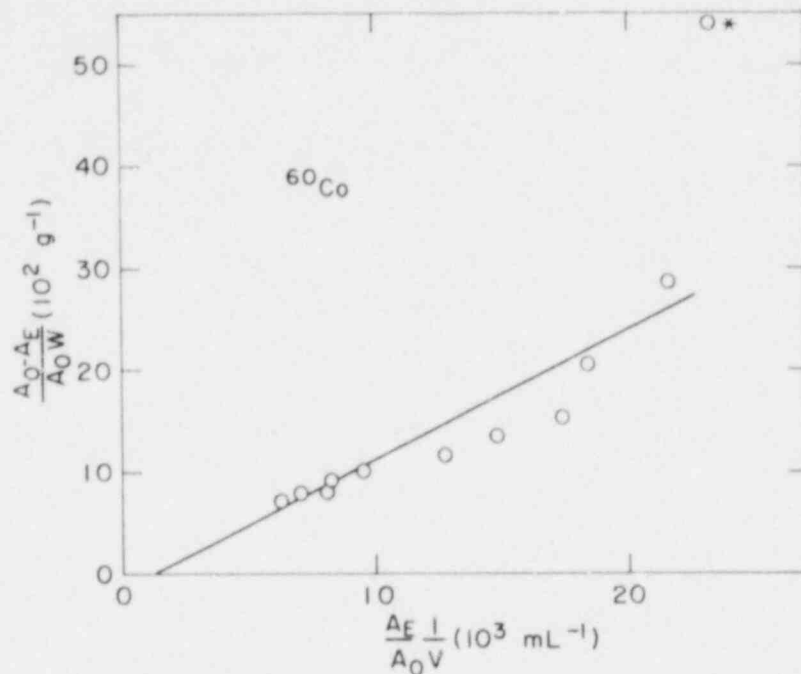


Figure 2.8 ^{60}Co sorption from Barnwell trench sump 6D1 water on Hawthorne sediment. The solid line represents the least squares linear regression. The data point indicated by * was not considered in the linear regression analysis.

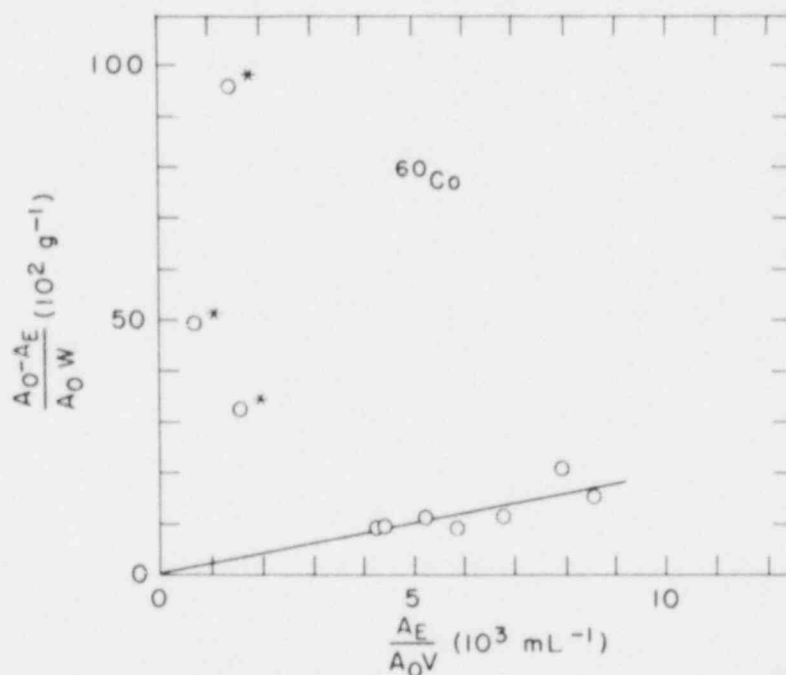


Figure 2.9 ^{60}Co sorption from Barnwell trench sump 25-21-D1 water on Hawthorne sediment. The solid line represents the least squares linear regression. The data points indicated by * were not considered in the linear regression analysis.

Table 2.3

Linear Sorption Isotherm Representation of Radionuclide Sorption Data for Barnwell Trench Sump 6D1 Water and Hawthorne Sediment

Isotope	Intercept ^a	Slope K _d (mL/g)	Correlation Coefficient
²⁴¹ Am ^b	0.008	1600	0.89
⁸⁵ Sr	0.008	13	0.99
¹³⁴ Cs	0.06	496	0.98
¹³⁷ Cs	0.06	486	0.97
⁶⁰ Co ^b	-0.02	13	0.99

^aTheoretical intercept = 0.

^bThe greatest solution/soil data point was neglected in the linear regression analysis.

Table 2.4

Linear Sorption Isotherm Representation of Radionuclide Sorption Data for Barnwell Trench Sump 25/21-D1 Water and Hawthorne Sediment

Isotope	Intercept ^a	Slope K _d (mL/g)	Correlation Coefficient
²⁴¹ Am ^b	0.1	86	0.79
¹³⁴ Cs	0.07	64	0.99
¹³⁷ Cs	0.06	65	0.99
⁶⁰ Co ^c	-0.03	21	0.81

^aTheoretical intercept = 0.

^bThe two greatest solution/soil ratio data points were neglected in the linear regression analysis.

^cThe three lowest solution/soil ratio data points were neglected in the linear regression analysis.

Table 2.5

Comparison of Batch, Column Loop and Isotherm K_d for Barnwell Trench Sump 6D1 Water and Hawthorne Sediment

Isotope	K_d (mL/g)		
	Batch ^a (mL/g)	Column Loop ^b (mL/g)	Isotherm (mL/g)
²⁴¹ Am	1150	>1200 ^c	1600
⁸⁵ Sr	14	35	13
¹³⁴ Cs	670	960	496
¹³⁷ Cs	670	960	486
⁶⁰ Co	18	1100 ^d	13

^aTrench 6D1 water (collected May 1980).

^bSynthetic 6D1 water and repacked Hawthorne sediment with a solution/soil ratio of 15:1 (Pietrzak et al., 1982).

^cBecause the activity in solution was only slightly above background, an accurate K_d could not be calculated.

^dA synthetic water was used in the column loop experiment. The presence of natural or synthetic complexing agents may account for the lower sorption results obtained by the batch and isotherm methods where the actual trench 6D1 water was used (Means et al., 1976).

Figures 2.4-2.7 show sorption isotherms for ¹³⁴Cs and ¹³⁷Cs. As expected, the ¹³⁴Cs and ¹³⁷Cs sorption coefficients are very similar (Tables 2.4 and 2.5). The Cs K_d values are approximately 490 mL/g for 6D1 water and 65 mL/g for 25/21-D1 water. Sokol (1970) reported ¹³⁷Cs K_d 's of 140 to 630 mL/g on silty sandstone which is comparable to the results for Barnwell materials. Table 3.5 shows results based on different methods. The values range from 490 mL/g for the sorption isotherm method to 960 mL/g for the column loop method.

⁶⁰Co sorption data are presented in Figures 2.8 and 2.9. Only minimum values of 13 mL/g and 21 mL/g were calculated for the distribution coefficient from the linear isotherm using the intermediate Co concentration data points (Tables 2.3 and 2.4). As indicated by the anomalous data points in Figures 2.8 and 2.9, the sorption relationship does not hold at low and high Co concentrations. Cobalt exhibits a rather complex sorption behavior. The soil components, Fe₂O₃ (Gonzalez and Gomez, 1964) and illite (Friend 1963, Sorathesn et al., 1960) have been shown to have a strong affinity for cobalt.

Between the solution pH of 6 to 9, cobalt sorption increases due to the formation of cobalt colloids (Sorathesn et al., 1960) and hydrolysis of Co^{+2} (Tewari et al., 1973). Cobalt is also relatively easily complexed by natural organics (Duguid, 1976; Means et al., 1976). As shown in Table 2.5, the ^{60}Co K_d values based on the batch and isotherm methods are comparable. However, the result, based on the column loop method where a synthetic water simulating Barnwell trench 6D1 water was used, was two orders of magnitude larger. The decreased sorption for the batch and isotherm method may be due to the presence of complexing agents in the natural trench water.

3. CHEMICAL CHANGES AND RADIONUCLIDE SCAVENGING DURING THE OXIDATION OF TRENCH WATER (M. Kinsley and J. Clinton)

3.1 Introduction

Changes in the redox state of disposal trench waters may affect radionuclide migration in groundwater systems. Waters collected from disposal trenches are generally anoxic, chemically-reducing solutions. Oxidation of anoxic trench waters results in significant changes in pH, Eh, alkalinity, and the solubilities of compounds. Iron, for example, is present in the soluble ferrous state in anoxic trench waters and, following oxidation, precipitates as ferric hydroxide. This precipitate may coprecipitate radionuclides and thereby provide a mechanism for retarding radionuclide migration.

Preliminary data on the oxidation of anoxic trench waters were reported earlier (Pietrzak et al., 1982). In this report, we present the results of laboratory experiments to study the chemical changes and the degree of coprecipitation that occur during the oxidation of Maxey Flats and West Valley trench waters.

3.2 Materials

Trench waters collected from trench 27 at Maxey Flats (MF27) in November 1979 and trench 8 in West Valley (WV8) in October 1978 were used in the experiments. The characteristics of these disposal trench waters have been reported previously (Czyscinski et al., 1981; Weiss and Colombo, 1980).

A mixed-radionuclide spike was prepared from high specific-activity radioisotopes. ^{241}Am , ^{134}Cs , ^{137}Cs , ^{85}Sr , and ^{60}Co radionuclides were mixed, evaporated to dryness, and dissolved in deionized water. Aliquots of the aqueous solution were used as reference solutions and as spikes for each oxidation experiment.

Radionuclide activities were determined by counting on a Ge(Li) detector with a multichannel analyzer system that was calibrated using a National Bureau of Standards mixed-radionuclide standard (NBS SRM 4242H25). Gamma-ray energies used in the determinations are listed in Table 3.1.

Table 3.1

Energies of the Gamma Photons Used
for Gamma Ray Analysis of the Radionuclides

Radionuclide	Gamma Ray Energy (keV)
²⁴¹ Am	59
⁶⁰ Co	1173, 1333
¹³⁴ Cs	605, 796
¹³⁷ Cs	662
⁸⁵ Sr	514

3.3 Experimental Procedures

The experiments were conducted in controlled environment chambers that were described in a previous report (Pietrzak et al., 1982). Trench water (about 650 mL) was anaerobically transferred from a collection bottle into a nitrogen-flushed, controlled-environment chamber containing a combination pH, a double-junction reference, and two platinum (one platinum for the carrier-free WV8 experiment) electrodes (Weiss and Colombo, 1980). The water was stirred continuously. The pH and Eh were monitored throughout the experiment. An initial sample was removed through the septum, filtered through a 0.45 μ m membrane (Gelman Acrodisc), and analyzed for alkalinity, ferrous iron, total iron, and background radionuclide activities. The alkalinity, ferrous iron and total iron (colorimetric) were determined using procedures described previously (Weiss and Colombo, 1980).

A known aliquot of a mixed spike, containing μ Ci amounts of each radionuclide, was added and the purge gas was stopped. The contents of the chamber slowly oxidized as air leaked into the chamber, resulting in a reddish-brown precipitate. Samples were removed periodically, filtered through 0.45 μ m membranes, and analyzed for radionuclides and ferrous and total iron. Total iron was determined in the filtrates from the carrier-free WV8 experiment using atomic absorption spectroscopy. Yields for ferrous and total iron were calculated from comparisons with the initial (pre-spiked) sample. Yields for Am, Co, Cs, and Sr were calculated from comparison with the reference aliquots of a mixed spike after correcting for any background nuclides detected in the initial sample. Cesium yields are means of yields determined from the net counts for the 605, 662, and 796 keV gamma ray peaks. Cobalt yields are means of yields calculated from the net counts for the 1173 and 1333 keV gamma-ray peaks.

The experiment was terminated when the total iron, radionuclide activities, Eh, and pH reached steady-state conditions. The contents of the chamber were filtered through a 0.45 μ m membrane filter and air dried. The precipitate was dissolved, analyzed for total iron by atomic absorption, and counted for specific gamma emitters.

Since cesium activities remained constant during the experiment, the total volume of trench water transferred to the chamber was calculated from the mean cesium activities in all the samples and the reference aliquots. This volume was compared with the volume calculated for the sum of volumes of the samples and the contents of the chamber at the time of filtration.

In the second of two experiments performed using trench water (WV8), sufficient $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ and CsCl carriers were added, prior to adding the mixed radionuclides, to obtain a concentration of 10^{-5} M for each metal in the trench water. Previous analyses (Weiss and Colombo, 1980) indicated concentrations of 10^{-6} M Cs and 2×10^{-5} M Sr in this trench water. The concentration of cobalt was not determined.

The precision for the ferrous iron and the total iron colorimetric analyses and for the total iron atomic absorption analysis are generally $\pm 3\%$ and $\pm 5\%$, respectively. Sufficient net counts (total counts corrected for continuum background) were generally accumulated in all gamma peaks to give $< \pm 3\%$ counting errors at the 95% confidence limit. The counting errors are larger ($< \pm 20\%$) for low Am yields due to the very high continuum to peak ratio for the low energy 59 keV photon. The spike added to the MF27 experiment contained a low concentration (approx. 0.1 μCi) of ^{85}Sr , therefore, the counting errors at the 95% confidence limit increased to $\pm 15\%$ for the 514-keV photon.

The total volumes in the chambers determined from the mean ^{137}Cs activities compared favorably with the volume summations. The errors for the activity method ($\pm 3\%$) were smaller than those for the summation method ($\pm 10\%$). Therefore, the activity method was used to determine the total volumes in the chambers.

3.4 Results and Discussion

The changes in Eh, pH, alkalinity, and metal concentrations before and after the oxidation of the WV8 and MF27 trench waters are reported in Table 3.2. The important differences in these trench waters are high alkalinity and relatively low iron concentration for WV8 and high iron concentration and low alkalinity for MF27. Also, MF27 has a lower initial redox potential than WV8.

The observed changes as a function of time are shown in Figure 3.1 for the Maxey Flats experiment and in Figures 3.2 and 3.3 for the West Valley experiments. The Eh values in Figures 3.1 and 3.3 represent average values based on readings from two electrodes and are precise to within ± 15 mV.

During the oxidation of the MF27 trench water (Figure 3.1), the most pronounced changes are observed in dissolved iron concentration, pH, and Eh during the first 25 hours, followed by a gradual change approaching equilibrium conditions. The ferrous iron concentrations range from 98.0 to 103% (mean 99.5%; 1 standard deviation = 1.2 for 14 samples) of the total dissolved iron concentrations indicating that all iron is present in the ferrous state. The yields for ferrous and total iron are $79 \pm 3.3\%$ for the last seven samples shown in Figure 3.1. Co, Cs, and Sr do not exhibit removal from solution.

Table 3.2

Changes in Alkalinity, Metal Concentrations, and Redox Potential Measured Before and After Oxidation of WV8 and MF27 Trench Waters

Component	Concentrations (moles/L) ^a					
	Maxey Flats Trench 27		West Valley Trench 8			
	Before	After	Carrier-Free Exp.		Carrier Exp. ^b	
	Before	After	Before	After	Before	After
Total Iron	2.7×10^{-2}	2.2×10^{-2}	2.0×10^{-3}	$< 6 \times 10^{-5}$	2.1×10^{-3}	4×10^{-6}
Ferrous Iron	2.7×10^{-2}	2.2×10^{-2}	f	f	2.1×10^{-3}	4×10^{-6}
Am ^c	7×10^{-9}	4×10^{-9}	6×10^{-9}	0	0	0
Cs ^d	$< 1 \times 10^{-6}$	$< 1 \times 10^{-6}$	1×10^{-6}	1×10^{-6}	1×10^{-5}	1×10^{-5}
Co ^e	$< 1 \times 10^{-5}$	$< 1 \times 10^{-5}$	f	f	1×10^{-5}	1×10^{-5}
Sr ^d	8×10^{-5}	8×10^{-5}	2×10^{-5}	2×10^{-5}	2×10^{-5}	2×10^{-5}
pH (unitless)	8.0	3.7	7.8	6.5	7.9	7.0
Eh (mV, NHE)	-400	+400	-100	+275	-150	+360
Alkalinity (meq/L)	3	0	f	f	44	f

^aExcept where units are specified.

^bSufficient $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ and CsCl salts were added to make the trench waters 10^{-5} M in cobalt and cesium.

^cCalculated from ^{241}Am activity.

^dBased on earlier work [Czyscinski et al., (1981); Weiss and Colombo (1980)].

^eConcentrations in the trench waters were not known.

^fAnalysis not performed.

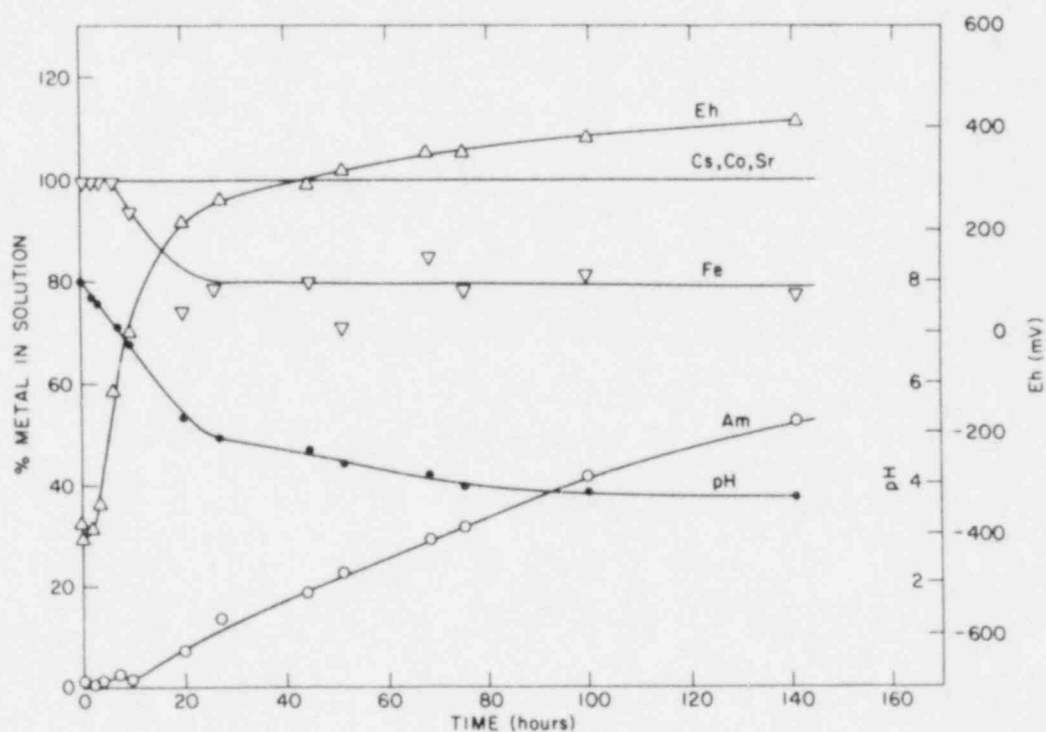


Figure 3.1 Changes in the pH, Eh and metal concentrations as a function of time during the oxidation of trench 27 water from the Maxey Flats disposal site.

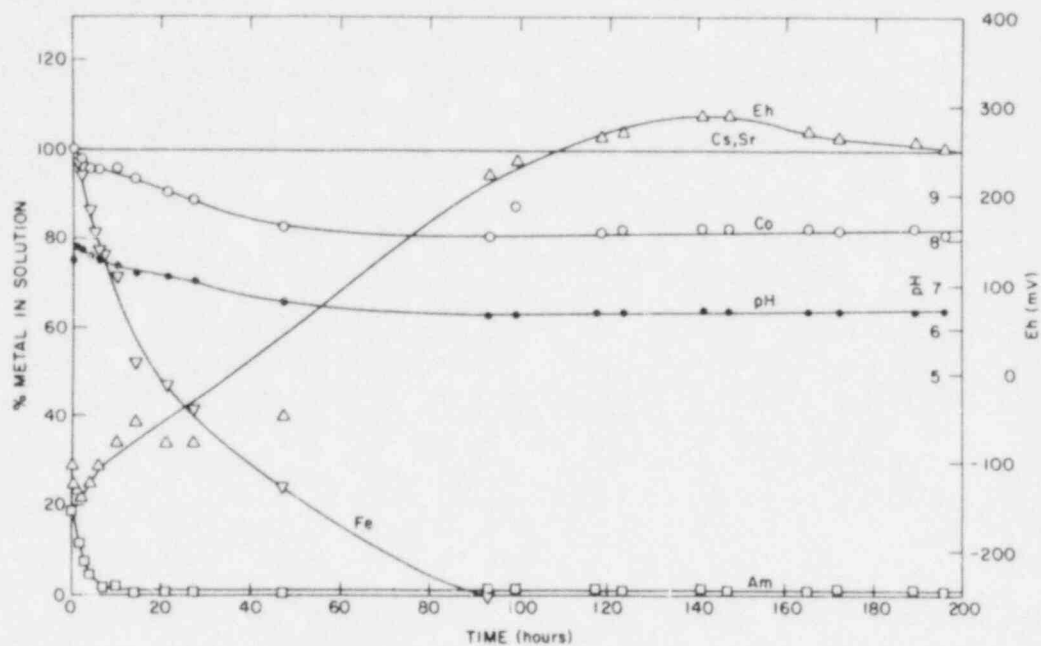


Figure 3.2 Changes in the pH, Eh and metal concentrations as a function of time during the oxidation of trench 8 water from the West Valley disposal site.

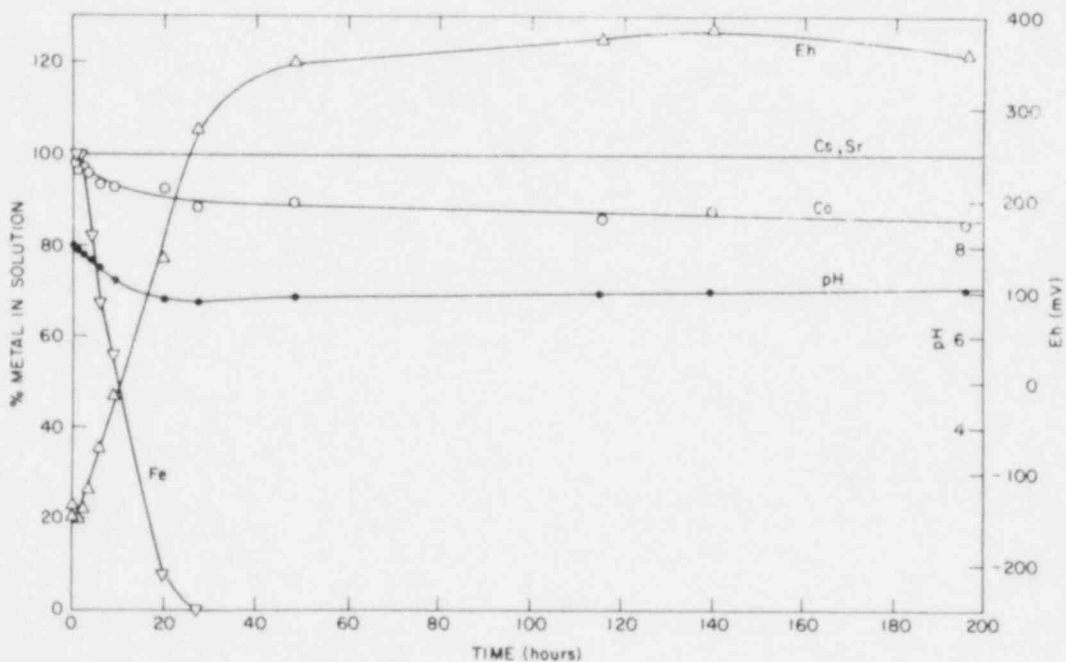


Figure 3.3 Changes in the pH, Eh, and metal concentrations as a function of time during the oxidation of trench 8 water from the West Valley disposal site with added $2 \times 10^{-5} \text{ M Cs}^+$ and Sr^{+2} .

Am concentration drops off sharply and remains low during the first 10 hours followed by a gradual increase. The Am behavior is believed to be a function of pH since the observations are in agreement with relative solubilities of americium oxides and hydroxides as influenced by pH (Ames and Rai, 1978). The relative solubilities are based on thermodynamic data and decrease rapidly with increasing pH. The yields for metals in the trench water and in the precipitate after steady-state conditions are reached are reported in Table 3.3. Small amounts of iron are lost on the 0.45 μ m membranes during the filtration of the aliquots removed from the chamber. No attempt was made to recover the iron from the membranes or the Am that is adsorbed on these iron precipitates or on the chamber walls.

Oxidation of the MF27 trench water beyond 140 hours indicates no further change in the metal concentrations. In addition, all the iron remained in the ferrous state even after prolonged bubbling of air through the filtrate (Table 3.4).

Table 3.3

Equilibrium Yield for Various Metals
in the Filtrate and Precipitate From
Oxidized Maxey Flats Trench 27 Water

Metal	Equilibrium Yield (%)		Total
	Precipitate	Filtrate	
Fe _{Total}	14	79 \pm 3 ^a	93
Am	29	62 \pm 2 ^b	91
Co	0.3	99 \pm 2 ^c	100
Cs	0.1	100 \pm 3 ^c	100
Sr	<0.08	110 \pm 14 ^c	110

^aMean and standard deviation for the last eight filtrates.

^bSee Table 3.4.

^cMean and standard deviation for 13 filtrates.

Table 3.4

Analyses of the Filtrate After Removal of the Ferric Oxyhydroxide
Precipitate From Maxey Flats Trench 27 Water

Component	Hours After Start of Experiment					\bar{x} ^a	+ <u>s</u> ^b
	914	1078	1249	1251	1509		
Am (%)	c	64	61	60	63	62	2
Cs (%)	c	100	100	105	100	100	3
Co (%)	c	105	104	104	105	104	1
Sr (%)	c	103	106	109	113	108	4
Fe ²⁺ (%)	d	79	81	81	80	80	1
Fe _{Total} (%)	c	80	82	81	80	80	1
Eh (mV)	+540	+540	+542	+546	a		
pH	3.1	3.0	2.8	2.9	a		

^a \bar{x} = Mean.

^bs = Standard deviation.

^cAnalysis not performed.

^dPipette malfunctioned.

The results in Figures 3.2 and 3.3 for the carrier-free and carrier experiments using trench 8 water from West Valley (WV8) indicate similar trends with time, but the rate of oxidation was faster in the carrier experiment probably due to greater air leakage into the chamber. Cs and Sr remained in solution while all the dissolved iron precipitated. The ratio of ferrous to total iron is 1.00 ± 0.02 for the first seven samples removed from the carrier-experiment chamber. The pH values stabilized between 6.5 and 7.0 and the Co concentrations stabilized at 83% and 88% for the carrier-free and carrier experiments, respectively. About 80% of the Am was removed from the solution during the first hour, followed by a gradual decrease until it reached about 1% after 10 hours. The concentrations of metals in the ferric oxyhydroxide precipitates and in the oxidized trench waters are listed in Table 3.5. Small amounts of ferric oxyhydroxide precipitates were lost on the membrane filters during the filtration of each aliquot removed from the chambers.

Table 3.5

Equilibrium Yield for Various Metals in the Filtrate
and Precipitate From Oxidized West Valley Trench 8 Water

Metal	Equilibrium Yield (%)					
	Carrier-Free Experiment			Carrier Experiment		
	Fe ppt	Filtrate	Total	Fe ppt	Filtrate	Total
Fe _{Total}	>97	<3	>97	96	0.2	96
Am	96	1.2+0.2 ^a	97	0	0	0
Cs	0.6	100.6+0.7 ^b	101	0.2	100.0+0.6 ^c	100
Co	18.0	83 ±2 ^a	101	12	88 ±1 ^c	100
Sr	1.8	99 ±3 ^b	101	2	98 ±1 ^c	100

^aMean and standard deviation for last 10 filtrates.

^bMean and standard deviation for 35 filtrates.

^cMean and standard deviation for 10 filtrates.

The iron concentrations as a function of Eh for the Maxey Flats and West Valley experiments are shown in Figure 3.4. Slight decreases in the dissolved iron concentrations occur between -100 and +200 mV in the Maxey Flats (MF 27) experiment. In the West Valley (WV 8) experiments, sharp decreases in dissolved iron concentrations were observed between approximately -140 and +135 mV for both the carrier-free and carrier systems. Since similar results were obtained for the two West Valley experiments, the iron concentrations appear to be a function of the Eh values.

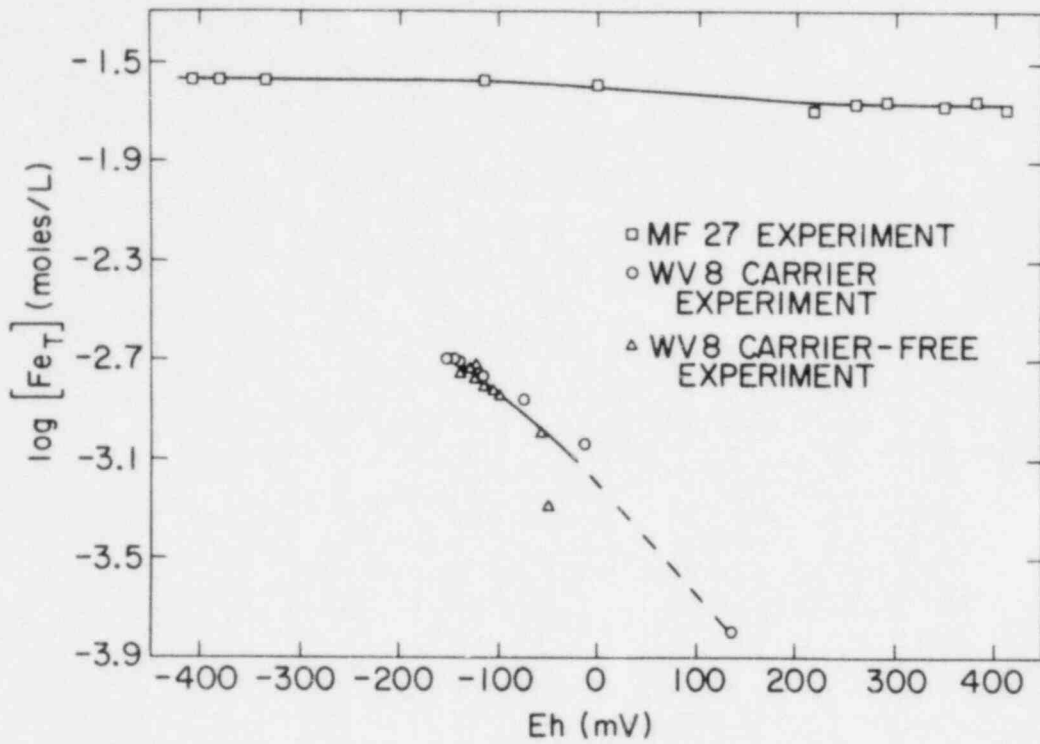


Figure 3.4 Dissolved iron concentrations as a function of Eh during the oxidation of trench waters from the Maxey Flats and the West Valley disposal sites. Dashed line represents extrapolation to the last data point.

4. AN INTEGRATED WASTE FORM/SEDIMENT LEACHING TEST (R. F. Pietrzak)

4.1 Introduction

Data on the leaching behavior of ion exchange resin and boric acid waste streams solidified in cement have been generated using a modified IAEA leach test at BNL (Morcos et al., 1982). Such tests are useful in making relative comparisons of waste forms and may represent a worst case condition in a water saturated environment.

A considerable amount of data on the chemical composition of disposal site trench waters have been generated at BNL (Pietrzak et al., 1982; Czyscinski et al., 1981; Weiss and Colombo, 1980). This information includes the concentrations of radionuclides, major and minor inorganic ions, pH, Eh, and conductivity. The trench water chemistry is important in establishing the source term useful in modeling the transport of contaminants by the groundwater. Laboratory experiments have also been conducted to determine the magnitude of radionuclide sorption using site specific materials. Radionuclide migration is controlled, to a large extent, by soil retardation properties and therefore this is an important consideration.

The scoping experiment proposed here is an integrated approach to approximate actual burial site conditions where, simultaneously, the leaching of the waste and radionuclide transport through the surrounding sediment is taking place in a partially water saturated environment. The waste form will be surrounded with Hawthorne sediment contained in an inert container. The waste form studied initially will consist of cation resin beads, loaded with ^{137}Cs and ^{85}Sr , solidified in Portland I cement. The waste-cement-water formulation used in making these forms will be identical to that used in our "scale-up" study (Morcos et al., 1982).

4.2 Materials

4.2.1 Waste Form

The composition of the waste form will be:

- Organic ion exchange resins: IRN-77, H^+ form, converted to the Na^+ form. Loaded with 100 μCi of ^{137}Cs and 200 μCi of ^{85}Sr .
- Cement: Portland I
- Waste to cement ratio: 0.6
- Water to cement ratio: 0.4
- Waste forms will be prepared in triplicate for each set of experimental conditions.

The nominal dimensions of the cylindrical waste form are 5 cm in diameter by 10 cm in height.

4.2.2 Leachant

A synthetic trench water simulating the composition of Barnwell trench 6D1 water will be used (Table 4.1). Preliminary experiments will be performed to determine what flow rate(s) will produce a uniform unsaturated continuous flow condition in the column without channeling or void formation through the sediment.

Table 4.1

Composition of Synthetic Barnwell Trench Water^a

Cations	Concentration mmoles/L	Anions	Concentration mmoles/L
Na ⁺	1.34	HCO ₃ ⁻¹	1.10
K ⁺	0.075	SO ₄ ⁻²	0.48
NH ₄ ⁺	0.26	Cl ⁻¹	0.38
Ca ⁺²	0.35	NO ₃ ⁻¹	0.30
Mg ⁺²	0.060		
Mn ⁺²	0.013		

SiO₂ = 0.12 mmoles/L^b

^aThe composition simulates water from trench sump 6D1 at Barnwell (Czyscinski and Weiss, 1981). The pH of the solution is adjusted to 6.0 with hydrochloric acid.

^bAdded as sodium metasilicate Na₂SiO₃·9H₂O.

4.2.3 Sediment

Sediment collected from the Hawthorne formation at the Barnwell site.

4.3 Apparatus

A column to hold the waste form and sediment will be constructed from polyethylene gallon jugs (15 cm in diameter by 30 cm in height). The waste form will be surrounded by 5 cm of sediment on each side. A plastic frit will be used to retain the sediment. Packing of the sediment in the column is critical to obtaining reproducible data. A fixed weight of sediment for each column will be uniformly packed with a plastic rod to a predetermined mark. A constant supply of leachant water will be available from a reservoir (see Figure 4.1). The metering plug in the leachant water reservoir will maintain continuous flow to assure unsaturated capillary flow through the sediment column.

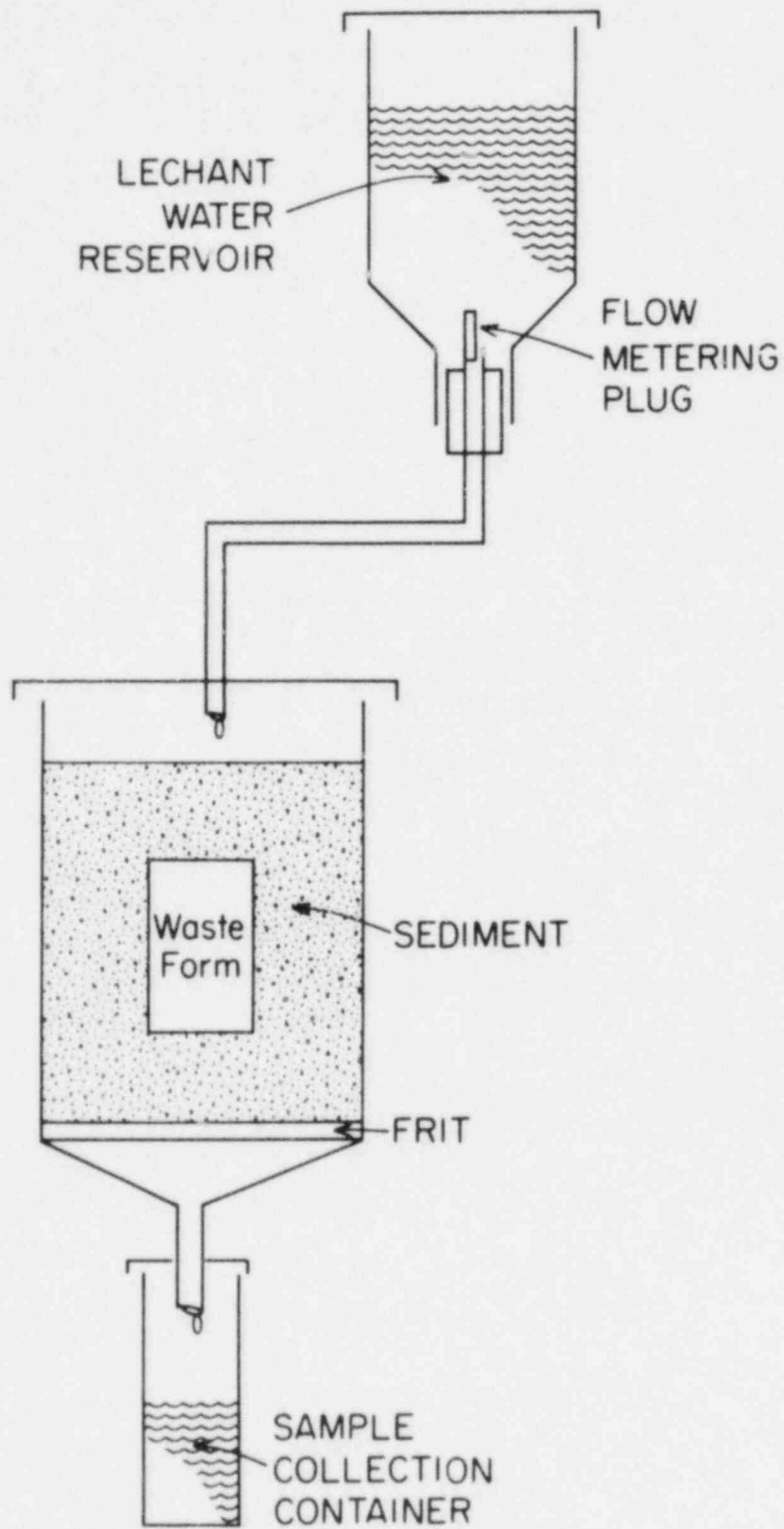


Figure 4.1 Apparatus for continuous capillary water flow leaching of solidified waste forms.

4.4 Leaching Procedure

The waste forms will be leached by allowing the leachant to percolate downward through the sediment at a continuous rate to simulate flow through the unsaturated zone. The activity in the eluate solutions will be determined using a Ge(Li) detector. Experiments will be conducted in triplicate for periods of 2, 4, 6 and 8 weeks.

The initial activity in the waste forms will be obtained by counting a sample of the radionuclide solution used to prepare the forms. At the termination of the experiment the activity remaining in the waste form will be determined by gamma counting a representative sample of the leached waste form. Based on the total activity measured in the eluate during the length of the experiment and the initial and residual activities in the waste form, the total amount of activity in the sediment will be determined.

4.5 Data Analyses

The data will be presented as the radionuclide fraction released vs time in a manner similar to the IAEA leaching procedure.

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