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Draft Phase II Results Report for Solvent Extraction Bench-Scale Treatability Study

Rocky Flats Plant

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Phase II Report

TABLE OF CONTENTS

Page

Table of Contents i

Table of Acronyms v

1.0 INTRODUCTION 1

1 1 Phase II Bench-Scale Program Purpose and Objectives . . 1

2.0 BENCH-SCALE TEST DESIGN 3

2 1 Sample Preparation 3

2 1 1 Soil Sample #1 Preparation 4

2 1 2 Soil Sample #2 Preparation 4

2 1 3 Vegetation Sample Preparation 4

2 1 4 Soil Particle Size Distribution Analytical Testing 5

2 2 Solvent-Extraction Tests . 5

2 2 1 Soil Sample #1 Solvent-Extraction Tests . . 6

2 2 2 Soil Sample #2 Solvent Extraction Tests . 6

2 2 3 Vegetation Sample Solvent Extraction Tests 7

3.0 ANALYTICAL AND QUALITY ASSURANCE/QUALITY CONTROL PROGRAM DESIGN 14

3 1 Analytical Program Design 14

3 1 1 Feed Sample Chemical Characterization . . 14

3 1 2 Radiochemical Analyses for Bench-Scale Solvent Extraction Tests 15

3 2 Quality Assurance/Quality Control Program 15

4.0 RESULTS AND DISCUSSION 17

4 1 Phase II Bench-Scale Testing . 17

4 1 1 Feed Sample Preparation Results 17

4 1 2 Soil Sample #1 Test Results 20

4 1 3 Soil Sample #2 Test Results 20

4 1 4 Vegetation Sample Test Results 21

4 2 Evaluation of Phase II Analytical Results 21

4 2 1 Statistical Evaluation of Feed Soil 21

4.2 2 Evaluation of Removal Efficiencies for Plutonium 23

Phase II Report

TABLE OF CONTENTS **Page**

(continued)

4 2 2 1 Soil Sample #1 Pu-239,240 Percent Removal Results 23
4 2 2 2 Soil Sample #2 Pu-239,240 Percent Removal Results 24
4 2 2 3 Vegetation Sample Pu-239,240 Percent Removal Results 24
4 2 3 Solids and Pu-239,240 Mass Balances 24
4 2 3 1 Soil Sample #1 Solids and Contaminant Mass Balance Results 25
4 2 3 2 Soil Sample #2 Solids and Contaminant Mass Balance Results 25
4 2 3 3 Vegetation Sample Solids and Contaminant Mass Balance Results 25

5 0 SUMMARY OF RESULTS AND RECOMMENDATIONS 38

List of Tables

1 1	Treatability Study Benchmarks	2
2 1	Summary of Solvent Extraction Test Parameters for Soil Sample #1	8
2 2	Summary of Solvent Extraction Test Parameters for Soil Sample #2	9
2 3	Summary of Solvent Extraction Test Parameters for Vegetation Sample	10
3 1	Summary of Phase II Sampling and Analytical Program	16
4 0	Summary of Sieve Test Results	19
4 1	Summary of Pu-239,240 Chemical Characterization Analytical Results	27
4 2	Summary of Soil Sample #1 Solids Analytical Results	28
4 3	Summary of Soil Sample #2 Solids Analytical Results	29
4 4	Summary of Vegetation Solids Analytical Results	30
4 5	Feed Soil and Vegetation Statistical Evaluation of Plutonium Analysis	31
4 6	Soil Sample #1 Pu-239,240 Removal	32
4 7	Soil Sample #2 Pu-239,240 Removal	33
4 8	Vegetation Sample Pu-239,240 Removal	34
4 9	Soil Sample #1 Solids and Pu-239,240 Mass Balance Results	35
4 10	Soil Sample #2 Solids and Pu-239,240 Mass Balance Results	36
4 11	Vegetation Sample Solids and Pu-239,240 Mass Balance Results	37
5 1	Summary of Phase II Bench-Scale Test Results	38

List of Figures

2 1 Soil Samples #1 and #2 Bench-scale Test and Analytical Sample Preparation 11
2 2 Vegetation Bench-scale Test and Analytical Sample Preparation 12
2 3 Bench-scale Solvent Extraction Process for Removal of Radionuclides 13

Appendixes

A Analytical Results
B Mass Balance Data

TABLE OF ACRONYMS

ARAR	Applicable or Relevant and Appropriate Requirement
ATI	Analytical Technologies, Inc
CFR	Code of Federal Regulations
cm	Centimeter
COCs	Contaminants of Concern
CTR	Contractors Technical Representative
FS	Feasibility Studies
HASP	Health and Safety Plan
HLA	Harding Lawson Associates
kg	Kilogram
ml	Milliliter
OU	Operable Unit
pCi/g	Picocuries per Gram
PPE	Personal Protective Equipment
PRG	Preliminary Remediation Goals
QAA	Quality Assurance Addendum
QAPP	Quality Assurance Project Plan
QC	Quality Control
RCC	Resources Conservation Company
RCRA	Resource Conservation and Recovery Act
RFP	Rocky Flats Plant
SHMP	Sodium hexametaphosphate
SOP	Standard Operating Procedure
TSBs	Treatability Study Benchmarks
F	Degrees Fahrenheit
g/g	Micrograms per gram

PHASE II REPORT

1 0 INTRODUCTION

This Draft Phase II Report has been prepared by Resources Conservation Company (RCC) as a contract deliverable between EG&G Rocky Flats Plant (RFP) (for the Environmental Restoration Group) and RCC. The purpose of this Draft Phase II Report is to describe the technical approach, results, and assessment of the solvent extraction bench-scale treatability study

1.1 Phase II Bench-Scale Program Purpose and Objectives

The purpose of the bench-scale treatability study is to provide data to support the Feasibility Studies (FS) in assessing the feasibility of using solvent extraction to remediate radionuclide-contaminated RFP soil and vegetation To fulfill the purpose of this treatability study, the following objectives were established for Phase II testing (1) confirm the reproducibility of the plutonium removal results achieved during Phase I testing; (2) evaluate the effect of additional extraction stages on removal of plutonium form RFP soil and vegetation beyond that tested in Phase I, (3) calculate the percent of Pu-239,240 removed from RFP soil and vegetation after solvent extraction testing, (4) calculate a plutonium (Pu-239,240) and solids mass balance for each of the solvent extraction tests performed during Phase II, and (5) evaluate the performance of Phase II bench-scale tests in removing COCs from the soil and vegetation samples to concentrations at or below the treatability study benchmarks (TSBs) shown in Table 1 1

Table 1.1: Treatability Study Benchmarks

Radionuclide Parameters	Concentration
Americium-241*	2 38 pCi/g
Plutonium-239,240*	3 65 pCi/g
Uranium (Total)*	144 pCi/g

pCi/g Picocuries per gram

* Programmatic Preliminary Remediation Goal (PRG) - Draft

2.0 BENCH-SCALE TEST DESIGN

This section describes the bench-scale test design for the Phase II solvent extraction treatability study The solvent extraction bench-scale tests were designed to identify the effectiveness of solvent extraction technology in removing COCs from RFP soil and vegetation The Phase II bench-scale tests consisted of two general tasks sample preparation and solvent extraction tests Sample preparation involved screening, blending, and splitting two separate soil samples into analytical samples and bench-scale test samples In addition, sample preparation involved cutting, blending, and splitting the vegetation sample into analytical samples and bench-scale test samples Sample preparation was performed prior to conducting Phase I testing The same feed material was used for both Phase I and Phase II testing

The solvent extraction tests were performed using triethylamine and those reagents and operating conditions identified in Phase I testing which yielded the most favorable plutonium removal The specific technical approach of the sample preparation and solvent extraction tests are described in the following sections

2.1 Sample Preparation

Soil and vegetation samples selected by EG&G from RFP were submitted to the laboratory for sample preparation, chemical characterization, and bench-scale testing The on-site laboratory prepared the soil samples by screening, blending, and splitting each sample into separate test and analytical samples. RCC technicians prepared the vegetation sample on-site by chopping, blending and splitting the vegetation sample into separate test and analytical samples The detailed descriptions of the sample preparation for soil sample #1, soil sample #2, and the vegetation sample are presented in the following sections These samples were prepared prior to conducting Phase I testing The prepared feed samples were used for both Phase I and Phase II testing

2.1.1 Soil Sample #1 Preparation

A standard Tyler sieve was used to remove material greater than 1/4 inch in diameter The amount of oversize material was recorded and the oversize material was set aside for return to RFP The blending process involved splitting the screened sample into two portions, recombining the split sample and mixing the recombined soil thoroughly The splitting, recombining and mixing (homogenization) step was performed a minimum of eight times Following the homogenization step, analytical and bench-scale test samples were divided as shown in Figure 2 1 The analytical samples were further divided into chemical characterization samples and submitted for chemical analysis The resulting analytical data on the feed provided the baseline to evaluate the effectiveness of the blending process and plutonium removal efficiencies during bench-scale testing

The bench-scale test sample portion selected for Phase II testing was homogenized again using the procedure described above An additional sample for feed characterization confirmatory analyses was withdrawn from the homogenized Phase II sample portion

2.1.2 Soil Sample #2 Preparation

Preparation of soil sample #2 consisted of screening, blending and splitting as described in Section 2 1 1 Following the screening and blending (homogenization) step, soil sample #2 was split into analytical and bench-scale test samples as shown in Figure 2 1 The analytical sample was further divided into chemical characterization samples and submitted for chemical analysis The resulting analytical data provided the baseline to evaluate the effectiveness of the blending process and plutonium removal efficiencies during bench-scale testing

The bench-scale test sample portion selected for Phase II testing was homogenized again using the procedure described above An additional sample for feed characterization confirmatory analyses was withdrawn from the homogenized Phase II sample portion

2.1.3 Vegetation Sample Preparation

The vegetation sample "as received" consisted of two root balls and their accompanying stems and leaves The stems and leaves were clipped with scissors and set aside The root balls were rinsed in a bucket of water to

remove any soil adhering to the surface of the vegetation Any floating material was skimmed off the water surface and set aside

The remaining root balls (minus the soil), stems, leaves, and skimmed material were combined and then ground in a meat grinder The resulting ground vegetation mixture was blended by hand using a split and combine technique as described in Section 2 1 1 Following the blending step, analytical and bench-scale test samples were split as shown in Figure 2 2 The analytical samples were further split and submitted for chemical analysis The resulting analytical data provided the baseline to evaluate the effectiveness of the blending process and plutonium removal efficiencies during bench-scale testing

The bench-scale test sample portion selected for Phase II testing was homogenized again using the procedure described above An additional sample for feed characterization confirmatory analyses was withdrawn from the homogenized Phase II sample portion

2.1.4 Soil Particle Size Distribution Analytical Testing

A sieve test was conducted on soil #1 and soil #2 to determine the distribution of material and activity as a function of particle size The sieve test was conducted using 150 grams of prepared feed soil. The feed soil was mixed with 150 ml of 0 1N sodium hexametaphosphate (SHMP) The SHMP acted as a dispersing agent for the soil slurry The soil slurry was then put through a series of screens arranged to capture sequentially smaller particle size fractions The three screens used were #5, #8 and # 40 mesh The material that passed through all screens was collected and flocculated to aid in separation of solids and liquid The liquid above the flocculated soil was decanted and filtered using a #40 and a #41 Whatman filter The flocculated solids were dried and combined with the material recovered via filtration and submitted to the laboratory for isotopic plutonium analysis The decanted liquid was collected after filtration and submitted for isotopic plutonium analysis

2.2 Solvent-Extraction Tests

Solvent-extraction tests were performed using the most effective combination of oxidizing/reducing agents, complexing agents, triethylamine, extraction time and extraction temperature identified during Phase I testing Generally, each extraction stage consisted of adding a reagent, mixing the sample with the reagent solution, separating liquids from solids (centrifugation) and recycling solids to the extraction vessel After completion of all the extraction stages, triethylamine was added to the separated reagent solution, contaminants were

concentrated, and water and triethylamine were recycled (Triethylamine was used to remove the water from the contaminant solution, allowing the water to be recycled without evaporation or other separation techniques) A block diagram of the solvent extraction bench-scale test process is presented in Figure 2 3

2.2.1 Soil Sample #1 Solvent-Extraction Tests

Two solvent extraction tests were conducted during Phase II testing of soil sample #1 The test parameters used for each test are presented Table 2 1 A step-by-step description of one of the two tests is given below to provide further clarification of the extraction sequence used during Phase II sample testing

A 200-gram portion of soil sample #1 was placed in a one-liter extraction vessel A solution of 3 percent hydrogen peroxide, used as an oxidizing agent, was added to the extraction vessel to achieve a liquid-to-solids ratio of 8 to 1 by weight. The solution was agitated and heated to 190°F Citric acid, used as a complexing agent, was then added to the extraction vessel to obtain a 0 1 molar citric acid solution The mixture was agitated for 60 minutes at 190°F and allowed to settle The solids were separated from the liquid extract solution via centrifugation This extract solution, free of suspended solids, was composited and later analyzed for isotopic plutonium, americium, and total uranium

The above extraction procedure was repeated eleven more times, starting with the addition of hydrogen peroxide to the solids and liquid remaining in the extraction vessel, for a total of twelve extraction stages The final treated solids were later analyzed for total uranium, isotopic plutonium, and americium After the final extraction, a composite extract solution was formed using the extract solution from each extraction stage Triethylamine was then added to the extract solution to concentrate the contaminants to a minimal volume (Addition of triethylamine forms a two-phase system a light phase containing triethylamine and water, and a heavy phase containing the contaminants and a small amount of water) The heavy phase was then analyzed for isotopic plutonium, americium, and total uranium The recovered water from extract solution concentration was sampled and later analyzed for isotopic plutonium, americium, and total uranium.

2.2.2 Soil Sample #2 Solvent Extraction Tests

Two solvent extraction tests were performed using the test parameters shown in Table 2 2 The step-by-step procedure used for soil sample #2 was similar to that discussed in Section 2 3.1 for soil sample #1.

2 2 3 Vegetation Sample Solvent Extraction Tests

One solvent extraction test was performed using the test parameters shown in Table 2 3 The step-by-step procedure used for the vegetation sample was similar to that discussed in Section 2 3 1 for soil sample #1

Table 2.1: Summary of Solvent Extraction Test Parameters for Soil Sample #1

Test No.	Oxidizing or Reducing Agent	Complexing Agent	Other Reagents Used	Number of Extraction Stages	Extraction Time Per Stage	Extraction Temperature
1	H_2O_2	$C_6H_8O_7$	Triethylamine	12	60 min	190 F
2	$Na_2S_2O_4$	$Na_3C_6H_5O_7$	Triethylamine	12	60 min	190 F

F	Degrees Fahrenheit
$C_6H_8O_7$	Citric acid
H_2O_2	Hydrogen peroxide
min	Minutes
$Na_3C_6H_5O_7$	Sodium citrate
$Na_2S_2O_4$	Sodium dithionite

Table 2.2: Summary of Solvent Extraction Test Parameters for Soil Sample #2

Test No	Oxidizing or Reducing Agent	Complexing Agent	Other Reagents Used	Number of Extraction Stages	Extraction Time Per Stage	Extraction Temperature
1	H_2O_2	$C_6H_8O_7$	Triethylamine	12	60 min	190 F
2	$Na_2S_2O_4$	$Na_3C_6H_5O_7$	Triethylamine	12	60 min	190 F

F	Degrees Fahrenheit
$C_6H_8O_7$	Citric acid
H_2O_2	Hydrogen peroxide
min	Minutes
$Na_3C_6H_5O_7$	Sodium Citrate
$Na_2S_2O_4$	Sodium dithionite

Table 2.3: Summary of Solvent Extraction Test Parameters for the Vegetation Sample

Test No.	Oxidizing or Reducing Agent	Complexing Agent	Other Reagents Used	Number of Extraction Stages	Extraction Time Per Stage	Extraction Temperature
1	$Na_2S_2O_4$	$Na_3C_6H_5O_7$	Triethylamine	12	60 min	190 F

F Degrees Fahrenheit
min Minutes
$Na_3C_6H_5O_7$ Sodium citrate
$Na_2S_2O_4$ Sodium dithionite

Figure 2 1? Soil feed split TREE

Figure 2 2 Veg feed split TREE

Figure 2 3 Block diagram of bench test process

3 0 ANALYTICAL AND QUALITY ASSURANCE/QUALITY CONTROL PROGRAM DESIGN

As described in Section 2 0, the sampling and analytical program was conducted in support of the Phase II solvent extraction bench-scale treatability study to assess the ability of solvent extraction technology to remove COCs from contaminated RFP soil and vegetation This analytical program was accompanied by a quality assurance/quality control (QA/QC) program to establish the defensibility of the analytical results The analytical and QA/QC programs conducted for the Phase II solvent extraction bench-scale treatability study are summarized below

3.1 Analytical Program Design

As described in Section 2 0, the analytical program for the Phase II bench-scale testing included two general components (1) chemical characterization of the RFP feed soil and vegetation samples prior to Phase I testing, and (2) radiochemical analyses for COCs in support of Phase II bench-scale solvent extraction tests Sample analyses for both components of the Phase II analytical program were performed at the laboratory on location

3.1.1 Feed Sample Chemical Characterization

Prior to conducting the Phase I bench-scale tests, the feed soil and vegetation samples obtained from RFP were screened, thoroughly blended, and split into analytical and test samples as described in Section 2 1 The analytical sample splits were submitted to the on-site laboratory, prior to Phase I testing, to be analyzed for the following radiochemical parameters Pu-238, Pu-239,240, Isotopic U, U (total), Am-241, gross alpha, and gross beta Plutonium and americium isotopes were analyzed for using alpha spectrometry, uranium was analyzed using kinetic phosphorimetry, and gross alpha and gross beta were analyzed using gas proportional counting The specific analytical protocols applied for the characterization of feed samples followed the on-site laboratory standard operating procedures (SOPs) for radiochemical analyses as documented in the Quality Assurance Addendum (QAA) for the solvent extraction bench-scale treatability study Results from the feed sample chemical characterization analyses established feed (influent) concentrations of COCs and were used to assess removals of COCs achieved by the Phase II tests A sample of the feed material used for Phase II testing was submitted to the laboratory for confirmatory analyses according to the procedures described above

In addition to radiochemical analyses, the Phase II analytical sample splits from the feed samples underwent screening analyses for total solids The total solids analyses were performed on location by RCC technicians using their internal SOPs

3.1.2 Radiochemical Analyses for Bench-Scale Solvent Extraction Tests

Extraction formulations and approaches which showed the greatest promise in Phase I testing based on percent plutonium removal results were repeated during Phase II testing Samples of major process media from these tests were submitted to the on-site laboratory to be analyzed for specific radiochemical parameters Process sample types and analytical parameters for these Phase II solvent extraction tests are summarized in Table 3 1 These analyses employed the same analytical methods used in the feed sample chemical characterization analyses, and followed the on-site laboratory's SOPs for radiochemical analyses as documented in the QAA for the solvent extraction bench-scale treatability study

3 2 Quality Assurance/Quality Control Program

QA/QC protocols were applied during the Phase II solvent extraction bench-scale treatability study to establish the reliability of the reported analytical results Multiple internal QC checks were performed during test sample analysis, including the analysis of duplicate test samples, laboratory method blanks, and laboratory control samples Radiochemical tracers were used as required by method SOPs to monitor the recovery of COCs from test sample matrices. Following sample analysis and results reporting, a data review and validation program was implemented to assess data quality and defensibility, and to identify potential limitations on data useability relative to the objectives of the treatability study This QA?QA review is still in progress and final results will be completed prior to submitting the final Phase I and II test report

Details of the QA/QC program for the solvent extraction bench-scale treatability study is provided in the QAA. Data quality issues identified from the application of QA/QC protocols in the Phase II testing will be discussed as they relate to specific test results in Section 4 0 A summary of overall data quality for the solvent extraction bench-scale treatability study will be included in the final report.

Table 3.1: Summary of Phase II Sampling and Analytical Program

Process Sample Type [a]	When Collected	Number of Samples Collected Per Test	Analytical Parameters		
			Pu-239,240	^{238}Pu	Total U
Interstage solids	After the 6th and 9th extraction stage	2	x	x	x
Interstage extract solution	Composite sample of the extract solution from extraction stages 1-6, and 7-12	2	x	x	x
Final treated solids	After final extraction stage	1	x	x	x
Final extract concentrate (heavy phase)	After combining and concentrating all extract solutions from test	1	x	x	x
Recovered Water	After combining and concentrating all extract solutions from test	1	x	x	x

Pu Plutonium
U Uranium

a Includes major process media sampled during Phase II tests

4.0 RESULTS AND DISCUSSION

This section presents the results of the Phase II bench-scale testing, as described in Section 2 0, including analytical results, percent contaminant removal calculations, mass balance calculations, and data quality assessment.

4.1 Phase II Bench-Scale Testing

The Phase II bench-scale tests produced both process and analytical results Process test results for each soil and vegetation sample included values for extraction temperature, extraction time, solids settling and centrifugation characteristics, oxidation/reduction agent addition, complexing agent addition, feed to reagent (i e , oxidation/reduction and complexing agent) ratios, and solvent to reagent ratios Analytical results identified the percent of plutonium removed for each set of process conditions for each soil and vegetation sample tested Analytical results provided data for mass balance calculations Analytical and test parameter results of the Phase I bench-scale testing were used to identify the test parameters to be used during Phase II testing Results from the two components of the bench-scale testing (sample preparation and solvent extraction tests) are summarized below for soil sample #1, soil sample #2, and the vegetation sample

4 1.1 Feed Sample Preparation Results

Following the screening, homogenization, and splitting described in Section 2 1, the chemical characterization analytical samples were submitted to the on-site laboratory for chemical analysis to provide data to evaluate the effectiveness of the blending process The results of plutonium chemical characterization analyses for each sample are presented in Table 4 1 Additional analytical results (i e , uranium, americium, and oil and grease, etc) for chemical characterization analyses for each sample are presented in Appendix A

A portion of the prepared feed material selected for each Phase II test was submitted to the analytical laboratory for confirmatory analyses The plutonium concentrations reported for soil sample #2 and the vegetation sample fell outside the 90% confidence interval determined during Phase I testing The analysis of these samples is currently being repeated. A discussion of these subsequent results will be provided in the final Phase I and II report

A sieve test was conducted on soil #1 and soil #2 as described in sections 2 1 4 and 4 1 1 The amount of material captured by each screen and the reported activity of each fraction is presented in Table 4 0 The

activity determination for the recovered liquid fraction is in progress and will be presented in the final Phase I and II report

Table 4.0: Summary of Sieve Test Results

	Dry wt., g	Activity pCi/g	
		Pu-239,240	Pu-238
Soil #1			
Feed soil used	138	740 *	--
Sieve fractions collected			
#5 mesh	1 0	16 7	< 0 425
#8 mesh	3 3	6 80	< 0 150
#40 mesh	65 5	72 0	1 20
< #40 mesh	69 7	312	5 30
Liquid	**	**	**
Soil #2			
Feed soil used	133	1350 *	--
Sieve fractions collected			
#5 mesh	0 3	15 8	< 0 315
#8 mesh	6 2	10 9	0 173
#40 mesh	40 0	23 7	0 41
< #40 mesh	86 9	679	11 3
Liquid	**	**	**

* Mean feed concentration

** Analysis in progress

4.1.2 Soil Sample #1 Test Results

The process data collected during Phase II bench-scale testing of soil sample #1 are summarized as follows

The extraction temperature was held constant at 190 F

The extraction time was 60 minutes per extraction stage

Solids settling times of up to 30 minutes were tested and centrifugation was required

The oxidation/reduction and complexing agents tested are presented in Table 2 1

The ratio of feed to reagent (i e , oxidation/reduction and complexing agents), expressed on a weight-to-weight basis, was 8 to 1

The ratio of solvent to reagent, expressed on a weight-to-weight basis, varied between 2 1 and 10 1

The results of Pu-239,240 and total uranium analyses conducted on soil sample #1 feed, interstage (i e , sixth and ninth extraction), and final treated solids from Phase II solvent extraction tests are presented in Table 4 3 The analytical results show that in tests 1 and 2, plutonium is reduced from a mean feed concentration of 740 pCi/g (as determined prior to Phase I testing) to 88 pCi/g and 83 pCi/g, respectively, in the final treated solids The concentration of plutonium in the solids is not significantly reduced after the initial four extractions. Therefore, conducting four extractions instead of twelve would have yielded similar results Additional analytical results (i e , extract solution, extract concentrate) from Phase II, soil sample #1 solvent extraction tests are presented in Appendix A

4.1.3 Soil Sample #2 Test Results

The process data collected during Phase II bench-scale testing of soil sample #2 are summarized as follows

The extraction temperature was held constant at 190 F

The extraction time was 60 minutes per extraction stage

Solids settling times of up to 30 minutes were tested and centrifugation was required

The oxidation/reduction and complexing agents tested are presented in Table 2 2

The ratio of feed to reagent (i e , oxidation/reduction and complexing agents), expressed on a weight-to-weight basis, was 8 to 1

The ratio of solvent to reagent, expressed on a weight-to-weight basis, varied between 2 1 and 10 1

The results of Pu-239,240 and total uranium analyses conducted on soil sample #2 feed, interstage (i e , sixth extraction, ninth extraction), and final treated solids from Phase II solvent extraction tests are presented in

Table 4 4 The analytical results show that in test 1, Pu-239,240 is reduced from a mean feed concentration of 1350 pCi/g (as determined prior to Phase I testing) to 100 pCi/g in the final treated solids The Pu-239,240 analysis of the treated solids for test 2 are under QA/QC review and will be discussed in the final Phase I and II test report The concentration of plutonium in the solids is not significantly reduced after the initial four extractions Therefore, conducting four extractions instead of twelve would have yielded similar results Additional analytical results (i e , extraction solution, extraction concentrate) from Phase II, soil sample #2 solvent extraction tests are presented in Appendix A

4.1.4 Vegetation Sample Test Results

The process data collected during Phase II bench-scale testing of the RFP vegetation sample are summarized as follows

The extraction temperature was held constant at 190 F

The extraction time was 60 minutes per extraction stage

Solids settling times of up to 30 minutes were tested and centrifugation was required

The oxidation/reduction and complexing agents tested are presented in Table 2 3

The ratio of feed to reagent (i e , oxidation/reduction and complexing agents), expressed on a weight-to-weight basis, was 8 to 1

The ratio of solvent to reagent, expressed on a weight-to-weight basis, was 2 1

The results of Pu-239,240 and total uranium analyses conducted on feed, interstage (i e , sixth extraction, ninth extraction), and final treated solids from Phase II solvent extraction vegetation testing are presented in Table 4 5 The analytical results show that in test 1, Pu-239,240 is reduced from a mean feed concentration of 620 pCi/g (as determined prior to Phase I testing) to 23 pCi/g in the final treated solids The concentration of plutonium in the solids is not significantly reduced after the initial four extractions Therefore, conducting four extractions instead of twelve would have yielded similar results Additional analytical results (i e , extraction solution, extraction concentrate) from Phase II, vegetation sample solvent extraction tests are presented in Appendix A

4.2 EVALUATION OF PHASE II ANALYTICAL RESULTS

Data evaluation of Phase II analytical results was performed to assist in assessing the overall bench-scale test performance during Phase II testing The following three specific evaluations of Phase II analytical data were performed (1) a statistical evaluation of feed soil analytical results (conducted prior to Phase I testing),

(2) evaluation of percent contaminant removal (Pu-239,240), and (3) evaluation solids and contaminant mass balances These evaluations are presented below

4.2.1 Statistical Evaluation of Feed Soil

For each of the two soil samples and one vegetation sample, the arithmetic mean, standard deviation, and 90 percent confidence intervals were calculated for the feed soil concentrations to assess the variability and homogeneity of the feed sample streams This statistical anlaysis was conducted prior to Phase I testing. These statistics were calculated for Pu-239,240 using the following formulas

$$\text{Mean} = \bar{x} = (\Sigma x_i) / n$$

$$\text{Standard deviation} = s = \sqrt{\frac{n\Sigma x_i^2 - (\Sigma x_i)^2}{n(n-1)}}$$

$$\text{Upper confidence interval} = (\bar{x}) + \frac{(t_{\alpha/2})(s)}{\sqrt{n}}$$

$$\text{Lower confidence interval} = (\bar{x}) - \frac{(t_{\alpha/2})(s)}{\sqrt{n}}$$

where

x_i = The concentration of Pu-239,240 measured in an individual replicate chemical characterization analysis of a feed sample during Phase II Three individual replicate analyses were performed for soil sample #1, and 4 such analyses were performed for soil sample #2 and the vegetation sample

n = The number of observations (i e , the number of individual replicate results for each soil or vegetation sample) For soil sample #1, n = 3, whereas n = 4 for soil sample #2 and the vegetation sample

$t_{\alpha/2}$ = The Student's t-Distribution for the 90 percent confidence interval for n-1 degree of freedom ($t_{\alpha/2}$ = 2 920 for 2 degrees of freedom [soil sample #1], and $t_{\alpha/2}$ = 2 353 for 3 degrees of freedom [soil sample #2 and the vegetation sample])

Statistics for the Phase I feed soil and vegetation samples are presented in Table 4 6 The same Phase I feed soil and vegetation was used for the Phase II tests Based on the range in values for the standard deviation, the statistics indicate that the highest degree of variability in feed soil results was observed in soil sample #2 and the vegetation sample results Specifically, the 90 percent confidence interval of the Pu-239,240 concentration in soil sample #2 ranged from 1113 pCi/g to 1585 pCi/g and from approximately 481 pCi/g to 752 pCi/g in the vegetation sample For these examples, a standard deviation of 201 pCi/g and 115 pCi/g was calculated for soil sample #2 and the vegetation sample, respectively, relative to mean concentrations of 1350 pCi/g and 620 pCi/g, respectively Calculation of percent relative standard deviations (% RSDs) based on these values ($S/\bar{x}$ times 100) produce % RSDs of less than 20 percent for both samples, which is considered acceptable for radiochemical analyses

Confirmatory feed analyses conducted during Phase II produced Pu-239,240 concentrations which were outside the 90% confidence interval for soil #2 and the vegetation These samples are being re-analyzed at this time and a discussion of the subsequent analysis results will be provided in the final Phase I and II report

4.2.2 Evaluation of Removal Efficiencies for Plutonium

The percentages of Pu-239,240 removed were calculated for Pu-239,240 for each of the solvent extraction tests conducted in Phase II The individual percentages of Pu-239,240 removed were calculated using analytical results from feed and treated solid samples as follows

$$\frac{\text{(Feed Sample Pu-239,240 Concentration)-(Treated Solid Pu-239,240 Concentration)} \times 100}{\text{(Feed Sample Pu-239,240 Concentration)}} = \text{Pu-239,240 Percent Removal}$$

The calculated results of the Pu-239,240 percent removal are presented below and are based on the mean feed concentrations calculated prior to conducting Phase I testing

4.2.2 1 Soil Sample #1 Pu-239,240 Percent Removal Results

The percentages of Pu-239,240 removed for Phase II testing of soil sample #1 are summarized in Table 4 7 The results for soil sample #1, based on the mean feed concentration, showed 88 and 89 percent removal of Pu-239,240 for tests number 1 and 2, respectively The parameters used during each of these tests are summarized in Table 2 4

4.2.2.2 Soil Sample #2 Pu-239,240 Percent Removal Results

The percentages of Pu-239,240 removed for Phase II testing of soil sample #2 are summarized in Table 4 8 The results for soil sample #2, based on the mean feed concentration, showed 92 percent removal of Pu-239,240 for test number 1 Incomplete QA/QC review of test results prevents calculation of percent removal for soil #2 The percent removal calculations for soil #2 will be discussed the final Phase I and II report The parameters used during each of these tests are summarized in Table 2 5

4.2.2.3 Vegetation Sample Pu-239,240 Percent Removal Results

The percentages of Pu-239,240 removed for Phase II testing of the vegetation sample is presented in Table 4 9. The results show a 96 percent removal of Pu-239,240 from the sample The parameters used during this test are summarized in Table 2 6

4.2.3 Solids and Pu-239,240 Mass Balances

Mass balance calculations were performed on the basis of Phase II test and analytical results Mass balance calculations consisted of solids mass balances and Pu-239,240 mass balances for each of the solvent extraction bench-scale tests conducted in Phase II Solids mass balances for each soil and vegetation sample were computed using the total weight of each feed sample, treated solids samples, in process samples, and nonvolatile reagents used during each solvent extraction test. Data used for calculation of the solids mass balances are presented in Appendix B The percent mass recovered for each test was calculated as follows

$$\text{Percent Solids Recovered} = \frac{\text{Mass of effluent solids}}{\text{Mass influent solids}} \times 100\%$$

where

Mass of effluent solids = total mass of treated solids recovered after completion of each solvent extraction test

Mass of influent solids = total mass of feed sample, on a dry basis, and mass of nonvolatile reagents

Contaminant mass balances for Pu-239,240 were conducted using the process and analytical data presented in Appendix A The percent contaminant recovery was calculated as follows

$$\text{Percent Pu-239,240 Recovered} = \frac{\Sigma\,[\text{Conc}_{(fr)} \times \text{Mass}_{(fr)}] \times 100\%}{(\text{Pu-239,240 Mass})_{(m)}}$$

where

$\text{Pu-239,240 Mass}_{(m)}$	=	$\text{Conc}_{(feed)}$ x Mass of feed sample in grams (as received basis)
$\text{Conc}_{(feed)}$	=	Mean concentration of Pu-239,240 in the feed sample, pCi/g (as received basis)
$\text{Conc}_{(fr)}$	=	Concentration (as received basis) of Pu-239,240 in the specific process fraction, pCi/g (this includes treated solids, interstage solids, and extract samples)
$\text{Mass}_{(fr)}$	=	Total (as received) mass of the specific process fraction, in grams

4.2.3.1 Soil Sample #1 Solids and Contaminant Mass Balance Results

Solids and Pu-239,240 mass balance results for soil sample #1 are summarized in Table 4 10 Solids mass balance results for soil sample #1 were 109 percent and 92 percent recovery for tests number 1 and 2, respectively Contaminant mass balance results for soil sample #1 show that the Pu-239,240 recoveries were 111 percent and 100 percent for test numbers 1 and 2, respectively The Pu-239,240 recoveries during Phase II testing of soil #1 were within the laboratory's control limits of 75 to 125 percent.

4.2.3 2 Soil Sample #2 Solids and Contaminant Mass Balance Results

Solids and Pu-239,240 mass balance results for soil sample #2 are summarized in Table 4 11 Solids mass balance results for soil sample #2 were 111 percent and 88 percent recovery for test numbers 1 and 2, respectively Contaminant mass balance results for soil sample #2 show that the Pu-239,240 recovery was 81 percent for test number 1 Incomplete QA/QC review of test results prevents calculation of percent recovery for soil #2 The percent recovery calculations for soil #2 will be discussed in the final Phase I and II report. The Pu-239,240 recovery was within the laboratory's control limits of 75 to 125 percent

4.2.3.3 Vegetation Sample Solids and Contaminant Mass Balance Results

Solids and Pu-239,240 mass balance results for the vegetation samples are summarized in Table 4 12 Solids mass balance results for the vegetation sample show 134 percent recovery Contaminant mass balance results for the vegetation sample show that the Pu-239,240 recovery was 148 percent. This recovery is outside the laboratory's control limit range of 75 to 125 percent The high recovery is attributed to extraction 1-6 composite extract sample non-homogeneity This will be confirmed by QA/QC data review and reanalysis, if required This data will be further discussed in the final Phase I and II test report.

Table 4.1: Summary of Pu-239,240 Chemical Characterization Analytical Results

	Soil Sample #1 (pCi/g)	Soil Sample #2 (pCi/g)	Vegetation Sample (pCi/g)
	NA	1530	681
	799	1514	599
	664	1200	463
	749	1152	725
Mean	740	1350	620
Phase II Confirmatory Analyses	688	895 *	340 *

pCi/g Picocuries per gram
NA Not analyzed
* Re-analysis underway, QA/QC Incomplete

Table 4.2: Summary of Soil Sample #1 Solids Analytical Results

	Results	
	Pu-230,240 (pCi/g)	**Total U (g/g)**
Test 1		
Feed*	740	12
6th ext. Solids	42	0 8
9th ext. Solids	44	0 9
12th ext. (final treated) Solids	88	0 9
Test 2		
Feed*	740	12
6th ext. Solids	32	1 1
9th ext. Solids	35	0 9
12th ext. (final treated) Solids	83	1 0

ext. Extraction
pCi/g Picocuries per gram
g/g Micrograms per gram

* Mean feed concentration (discussed in Section 4 2 1)

Table 4.3: Summary of Soil Sample #2 Solids Analytical Results

	Results	
	Pu-239,240 (pCi/g)	**Total U (g/g)**
Test 1		
Feed*	1350	6.2
6th ext. Solids	97	2 2
9th ext. Solids	108	2 1
12th ext. (final treated) Solids	102	2 5
Test 2		
Feed*	1350	6 2
6th ext. Solids	43	1 1
9th ext. Solids	278	1 7
12th ext. (final treated) Solids	#	2 7

ext. Extraction
pCi/g Picocuries per gram
g/g Micrograms per gram

* Mean feed concentration (discussed in Section 4 2 1)
\# Re-analysis in progress, QA/QC Incomplete

Table 4.4: Summary of Vegetation Sample Solids Analytical Results

	Results	
	Pu-239,240 (pCi/g)	**Total U (g/g)**
Test 1		
Feed*	620	5 7
6th ext. Solids	17	0.74
9th ext. Solids	19	0 80
12th ext. (final treated) Solids	23	0 82

ext. Extraction
pCi/g Picocuries per gram
g/g Micrograms per gram

* Mean feed concentration (discussed in Section 4 2 1)

Table 4.5: Feed Soil and Vegetation Statistical Evaluation of Plutonium Analysis

	Concentration		
	Chemical Characterization Results Soil Sample #1 (pCi/g)	**Chemical Characterization Results Soil Sample #2 (pCi/g)**	**Chemical Characterization Results Vegetation Sample (pCi/g)**
Concentrations	NA	1530	681
	799	1514	599
	664	1200	463
	749	1153	725
Mean	740	1350	620
Standard Deviation	68	201	115
90% Confidence Interval			
Upper Interval	852	1585	752
Lower Interval	622	1113	481

NA Not analyzed
pCi/g Picocuries per gram
% Percent

Table 4 6: Soil Sample #1 Pu-239,240 Removal

Test Number	Number of Extractions	Feed* Concentration (pCi/g)	Treated Solids Concentration (pCi/g)	Percent Removal
1	12	740	88	88
2	12	740	83	89

pCi/g Picocuries per gram

* Mean feed concentration (discussed in Section 4 2 1)

Table 4.7: Soil Sample #2 Pu-239,240 Removal

Test Identification	Number of Extractions	Feed* Concentration (pCi/g)	Treated Solids Concentration (pCi/g)	Percent Removal
1	12	1,350	102	92
2	12	1,350	#	#

pCi/g Picocuries per gram

* Mean feed concentration (discussed in Section 4 2 1)
\# Not available, QA/QC incomplete

Table 4.8: Vegetation Sample Pu-239,240 Removal

Test Identification	Number of Extractions	Feed* Concentration (pCi/g)	Treated Solids Concentration (pCi/g)	Percent Removal
1	12	620	23	96

pCi/g Picocuries per gram

* Mean feed concentration (discussed in Section 4 2 1)

Table 4.9: Soil Sample #1 Solids and Pu-239,240 Mass Balance Results

Test Number	Solids Recovery (percent)	Pu-239,240 Recovery* (percent)
1	109	111
2	92	100

pCi/g Picocuries per gram

* Based on mean feed concentrations

Table 4.10: Soil Sample #2 Solids and Pu-239,240 Mass Balance Results

Test Identification	Solids Recovery (percent)	Pu-239,240* Recovery (percent)
1	111	81
2	88	#

* Based on mean feed concentrations
\# Not available, QA/QC incomplete

Table 4.11* Vegetation Sample Solids and Pu-239,240 Mass Balance Results

Test Identification	Solids Recovery (percent)	Pu-239,240* Recovery (percent)
1	134	148

* Based on mean feed concentrations

5.0 SUMMARY OF RESULTS AND RECOMMENDATIONS

This section summarizes the RFP soil and vegetation Phase II bench-scale test results and provides recommendations based on these results Phase II bench-scale solvent extraction tests were evaluated to (1) confirm the reproducibility of the plutonium removal results achieved during Phase I testing, (2) evaluate the effect of additional extraction stages on removal of plutonium form RFP soil and vegetation beyond that tested in Phase I, (3) calculate the percent of Pu-239,240 removed from RFP soil and vegetation after solvent extraction testing, (4) calculate a plutonium (Pu-239,240) and solids mass balance for each of the solvent extraction tests performed during Phase II, and (5) evaluate the performance of Phase II bench-scale tests in removing COCs from the soil and vegetation samples to concentrations at or below the treatability study benchmarks (TSBs)

The results of the Phase II bench-scale tests, as summarized in Table 5 1, indicate that a significant removal of Pu-239,240 from RFP soil and vegetation was achieved during Phase II testing However, these removals were comparable to those achieved during Phase I testing, indicating that the majority of the plutonium removal occurred in the first 3-4 extraction stages The Pu-239,240 removal for the tests with the most favorable results was 89 percent for soil sample #1, 92 percent for soil sample #2, and 96 percent for the vegetation sample, based on the mean feed concentrations calculated prior to Phase I testing Pu-239,240 and solids mass balance results, summarized in Table 5 1, support the Pu-239,240 removal results. Solids recovery ranged from 88 to 134 percent and Pu-239,240 recovery ranged from 81 to 148 percent.

Based on the results of the Phase II bench-scale solvent extraction testing, it is evident that virtually all plutonium extraction took place during the first four extraction stages and the final eight extraction stages accomplished only limited additional extraction Removal of residual plutonium from the treated solids to below the TSBs would require further optimization testing to identify the optimum extraction parameters

Table 5.1. Summary of Phase II Bench-Scale Test Results

Sample	Number of Extractions	Pu-239,240 Removal (percent)	Pu-239,240 Recovery (percent)	Solids Recovery (percent)
Soil #1				
Test #1	12	88	111	109
Test #2	12	89	100	92
Soil #2				
Test #1	12	92	81	111
Test #2	12	#	#	88
Vegetation	12	96	148	134

Not available, QA/QC incomplete

Appendix A

Analytical Results

PHASE II

Sample ID	Description	Test No.	Analyses Results Pu-239/240,	Pu-238, pCi/g	Am-241, pCi/g	KPA U ug/g
Soil Sample Number One						
94-10-169						
-01	Untreated Feed	1&2	688	11 5	152 02	8 54
-02	6th Extraction Solids	1	41 8	0 757	8 712	0 75
-03	9th Extraction Solids	1	44 2	0 764	9 4248	0 9
-04	Composite Extract 1-6	1	14 3	0 242	2 421	166 53
-05	Composite Extract 7-12	1	0 146	< 0 014	0 035	1 63
-06	Treated Solids	1	88 3	1 63	18 968	0 89
-07	6th Extraction Solids	2	32 4	0 577	7 054	1 12
-08	9th Extraction Solids	2	35 4	0 651	7 464	0 93
-09	Composite Extract 1-6	2	13 3	0 236	2 296	183 61
-10	Composite Extract 7-12	2	0 335	< 0 022	0 085	1 78
-11	Treated Solids	2	83 3	1 46	16 572	0 99
-12	Recovered Water	1	0 605	< 0 022	0 132	8 42
-13	Concentrated Contaminant	1	153	2 63	27 829	1 43
-14	Recovered Water	2	0 041	< 0 048	< 0 011	8 01
-15	Concentrated Contaminant	2	42 7	0 734	7 582	57 59
Soil Sample Number Two						
94-10-233						
-01	Untreated Feed	1&2	707	11 7	192 68	6 36
-02	6th Extraction Solids	1	97 1	1 72	20 658	2 17
-03	9th Extraction Solids	1	108	1 97	21 297	2 059
-04	Treated Solids	1	102	1 84	22 408	2 48
-05	Composite Extract 1-6	1	18 6	0 317	3 254	0 103
-06	Composite Extract 7-12	1	0 298	< 0 005	?	0 001
-07	Recovered Water	1	0 113	< 0 011	0 023	0 001
-08	Concentrated Contaminant	1	26 4	0 441	5 788	0 083
-09	6th Extraction Solids	2	42 8	< 0 736	12 333	1 11
-10	9th Extraction Solids	2	278	4 68	71 315	1 72
-11	Treated Solids	2	355	6 14	80 73	2 72
-12	Composite Extract 1-6	2	16 6	0 256	2 912	0 103
-13	Composite Extract 7-12	2	0 911	0 015	0 379	0 003
-14	Recovered Water	2	0 69	0 013	0 131	0 016
-15	Concentrated Contaminant	2	37 8	0 562	6 978	0 182
-16	Rag Layer	2	48 1	0 738		
Vegetation Sample						
94-10-298						
-01	Feed	1	370	5 68	130 19	5 48
-02	6th Extraction Solids	1	16 6	< 0 492	4 109	0 74
-03	9th Extraction Solids	1	19 2	0 392	4 238	0 8
-04	Composite Extract 1-6	1	5 21	0 072	1 007	0 019
-05	Composite Extract 7-12	1	0 113	< 0 012	< 0 040	0
-06	Treated Solids	1	22 6	0 381	5 829	0 82
-07	Recovered Water	1	0 234	< 0 08	0 074	0 001
-08	Concentrated Contaminant	1	7 29	0 111	1 57	0 016
-09	Rag Layer	1	10 5	0 164	1 535	0 034

Appendix B

Mass Balance Data

Solids Mass Balances

Soil No 1, Citrate/Dithionite Test

Item Description	Mass,g	Total Solids, %	Net Solids, g
SOLIDS INPUT			
Feed	200	92 0%	184
Total Input			184
SOLIDS OUTPUT			
6 Solids	7	100 0%	7
9 Solids	8 7	100 0%	9
Extract Sol'n TSS	9446 7	0 1%	13
Extract Sol'n TSS	9640 1	0 1%	13
Treated Solids	159 2	100 0%	159
Total Output			200

TOTAL INPUT	184
TOTAL OUTPUT	200
PERCENT RECOVERY	**109.0%**

Soil No 1, Citric Acid/Peroxide Test

Item Description	Mass,g	Total Solids, %	Net Solids, g
SOLIDS INPUT			
Feed	200	92 0%	184
Total Input			184
SOLIDS OUTPUT			
6 Solids	9	100 0%	9
9 Solids	10 8	100 0%	11
Extract Sol'n TSS	9081 3	0 0%	2
Extract Sol'n TSS	9242 8	0 0%	3
Treated Solids	144 4	100 0%	144
Total Output			169

TOTAL INPUT	184
TOTAL OUTPUT	169
PERCENT RECOVERY	**91.8%**

Soil No 2, Citrate/Dithionite Test

Item Description	Mass,g	Total Solids, %	Net Solids, g
SOLIDS INPUT			
Feed	200	89 0%	178
Total Input			178
SOLIDS OUTPUT			
6 Solids	6 6	100 0%	7
9 Solids	10 3	100 0%	10
Extract Sol'n TSS	9459 7	0 1%	14
Extract Sol'n TSS	9585 8	0 2%	19
Treated Solids	148 1	100 0%	148
Total Output			197
TOTAL INPUT	178		
TOTAL OUTPUT	197		
PERCENT RECOVERY	**110.7%**		

Soil No 2, Citric Acid/Peroxide Test

Item Description	Mass,g	Total Solids, %	Net Solids, g
SOLIDS INPUT			
Feed	200	89 0%	178
Total Input			178
SOLIDS OUTPUT			
6 Solids	5 5	100 0%	6
9 Solids	7 3	100 0%	7
Extract Sol'n TSS	9671 5	0 1%	7
Extract Sol'n TSS	9652 4	0 0%	4
Treated Solids	133 3	100 0%	133
Total Output			157
TOTAL INPUT	178		
TOTAL OUTPUT	157		
PERCENT RECOVERY	**88.4%**		

Vegetation, Citrate/Dithionite Test

Item Description	Mass,g	Total Solids, %	Net Solids, g
SOLIDS INPUT			
Feed	100	29 0%	29 0
Reagent	78 5	4 6%	3 6
Total Input			32 6
SOLIDS OUTPUT			
6 Solids	1 5	100 0%	1 5
9 Solids	2 2	100 0%	2 2
Extract Sol'n TSS	4876 8	0 2%	8 6
Extract Sol'n TSS	4891 2	0 1%	6 4
Treated Solids	25	100 0%	25 0
Total Output			43 7
TOTAL INPUT	32 6		
TOTAL OUTPUT	44		
PERCENT RECOVERY	**134.0%**		

Phase II Plutonium-239/240 Mass Balances

Soil No. 1, Citrate/Dithionite Test

Item Description	Mass,g	Concentration, pCi/g	Net Activity, pCi
ACTIVITY INPUT			
Feed	184	740	136160
Total Input			136160
ACTIVITY OUTPUT			
Treated Solids	159 2	88 3	14057
6th Extraction Solids	7	41 8	293
9th Extraction Solids	8 7	44 2	385
Composite Extract (1-6)	9446 7	14 3	135088
Composite Extract (7-12)	9640 1	0 146	1407
Total Output			151230
TOTAL INPUT	136160		
TOTAL OUTPUT	151230		
PERCENT RECOVERY	**111.1%**	Percent Removal	88 1%

Soil No 1 Total Solids = 92%

Soil No 1, Citric Acid/Peroxide Test

Item Description	Mass,g	Concentration, pCi/g	Net Activity, pCi
ACTIVITY INPUT			
Feed	184	740	136160
Total Input			136160
ACTIVITY OUTPUT			
Treated Solids	144 4	83 3	12029
6th Extraction Solids	9	32 4	292
9th Extraction Solids	10 8	35 4	382
Composite Extract (1-6)	9081 3	13 3	120781
Composite Extract (7-12)	9242 8	0 335	3096
Total Output			136580
TOTAL INPUT	136160		
TOTAL OUTPUT	136580		
PERCENT RECOVERY	**100.3%**	Percent Removal	88 7%

Soil No 1 Total Solids = 92%

52

Soil No. 2, Citrate/Dithionite Test

Item Description	Mass,g	Concentration, pCi/g	Net Activity, pCi
ACTIVITY INPUT			
Feed	178	1350	240300
Total Input			240300
ACTIVITY OUTPUT			
Treated Solids	148 1	102	15106
6th Extraction Solids	6 6	97 1	641
9th Extraction Solids	10 3	108	1112
Composite Extract (1-6)	9459 7	18 6	175950
Composite Extract (7-12)	9585 8	0 298	2857
Total Output			195666
TOTAL INPUT	240300		
TOTAL OUTPUT	195666		
PERCENT RECOVERY	**81.4%**	Percent Removal	92 4%

Soil No 2 Total Solids = 89%

Soil No. 2, Citric Acid/Peroxide Test

Item Description	Mass,g	Concentration, pCi/g	Net Activity, pCi
ACTIVITY INPUT			
Feed	178	1350	240300
Total Input			240300
ACTIVITY OUTPUT			
Treated Solids	133 3	355	47322
6th Extraction Solids	5 5	42 8	235
9th Extraction Solids	7 3	278	2029
Composite Extract (1-6)	9671 5	16 6	160547
Composite Extract (7-12)	9652 4	0 911	8793
Total Output			218927
TOTAL INPUT	240300		
TOTAL OUTPUT	218927		
PERCENT RECOVERY	**91.1%**	Percent Removal	73 7%

Soil No 2 Total Solids = 89%

Vegetation, Citrate/Dithionite Test

Item Description	Mass,g	Concentration, pCi/g	Net Activity, pCi
ACTIVITY INPUT			
Feed	29	620	17980
Total Input			17980
ACTIVITY OUTPUT			
Treated Solids	25	22 6	565
6th Extraction Solids	1 5	16 6	25
9th Extraction Solids	2 2	19 2	42
Composite Extract (1-6)	4876 8	5 21	25408
Composite Extract (7-12)	4891 2	0 113	553
Total Output			26593
TOTAL INPUT	17980		
TOTAL OUTPUT	26593		
PERCENT RECOVERY	**147.9%**	Percent Removal	96 4%

Vegetation Total Solids = 29%