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DIST	LTR	ENC
AMARAL M E		
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BRANCH D B		
CARNIVAL G J		
DAVIS J G		
FERRERA D W		
FRAY R E		
GEIS J A		
GLOVER W S		
GOLAN P M		
HANNI B J		
HARMAN L K		
HEALY T J		
HEDAHL T		
HILBIG J G		
HUTCHINS N M		
JACKSON D T		
KELL R E		
KUESTER A W		
MARX G E		
McDONALD M M		
McKENNA F G		
MONTROSE J K		
MORGAN R V		
POTTER G L		
PIZZUTO V M		
RISING T L		
SANDLIN N B		
SCHWARTZ J K		
SETLOCK G H		
STEWART D L		
STIGER S G	X	
TOBIN P M		
VOORHEIS G M		
WILSON J M		
Schuh, D	L	
Long, A	X	
Foor, L	X	
CORRES CONTROL	X	X
ADMN RECORD/080	X	X
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EG&G ROCKY FLATS

EG&G ROCKY FLATS, INC
ROCKY FLATS PLANT, P O BOX 464, GOLDEN, COLORADO 80402-0464 • (303) 966-7000

November 4, 1994 94-RF-11144

Norma I Castaneda
Environmental Restoration Division
DOE/RFFO

SUBMITTAL OF SOLVENT EXTRACTION TREATABILITY STUDY (TS) PHASE I REPORT - WSB-123-94

Attention M J Harris

Action None

This correspondence serves as transmittal of the Solvent Extraction Phase I Treatability Study Progress Report Twelve copies of this report will be delivered to your office on Nov 7, 1994 One copy is being sent through the Department of Energy Rocky Flats Field Office (DOE/RFFO) mail room This report covers progress through September 8, 1994

As you know, the regulatory agencies have directed the Department of Energy Rocky Flats Field Office and EG&G Rocky Flats, Inc to provide them with treatability study results from the Sitewide Treatability Studies Program no later than November 12, 1994 This Phase I report could be used to satisfy that requirement

Please contact W J Roushey of the Treatability Studies/Feasibility Studies Team at extension 6951 or pager 1711

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ADMIN RECORD

SW-SW-A-02993

RFP/ERM-94-00032

Phase I Report for Solvent Extraction Bench-Scale Treatability Study

Rocky Flats Plant

U.S. Department of Energy
Rocky Flats Field Office
Golden, Colorado

Environmental Restoration Program

October 1994

Phase I Report

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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
SOP	Standard Operating Procedure
TSBs	Treatability Study Benchmarks
°F	Degrees Fahrenheit
µg/g	Micrograms per gram

PHASE I REPORT

1 0 INTRODUCTION

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

1 1 Phase I 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 I testing (1) generate performance data for removing contaminants of concern (COCs) from contaminated RFP soil and vegetation, (2) identify near optimum operating parameters (i e , number of extraction stages, extraction temperature, pH, solvent ratios, and pretreatment requirements) for removing COCs from contaminated RFP soil and vegetation using triethylamine, (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 I, and (5) evaluate the potential of Phase II bench-scale testing to remove 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 I 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 I bench-scale tests consisted of three general components sample preparation, screening tests 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 Screening tests were then performed to identify oxidizing, reducing, and complexing agents or other potential solubilizing agents showing the highest plutonium removal efficiencies for both soil and vegetation samples The solvent extraction tests were performed using triethylamine and those reagents identified in the screening test (that showed removal of 20 percent or greater Pu-239,240) to further evaluate solvent extraction's ability to remove COCs The specific technical approach of the sample preparation, screening tests, 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 Bench-testing technicians prepared the vegetation sample by blending and splitting the vegetation sample into separate test and analytical samples The detailed description of the sample preparation for soil sample #1, soil sample #2, and the vegetation sample are described in the following sections

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 provided baseline (feed) chemical data to evaluate the effectiveness of the blending process and plutonium removal efficiencies during bench-scale testing

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 baseline chemical data to evaluate the effectiveness of the blending process and plutonium removal efficiencies during bench-scale testing

2 1 3 Vegetation Sample Preparation

The "as received" vegetation sample 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 baseline chemical data to evaluate the effectiveness of the blending process and plutonium removal efficiencies during bench-scale testing

2.2 Screening Tests

Screening tests were performed on each soil and vegetation sample to evaluate plutonium removal using several combinations of oxidizing, reducing, and complexing agents or other potential solubilizing agents Each screening test consisted of adding one or more reagents and conducting one extraction stage Gross alpha screening was performed on the extract solution after each reagent addition to evaluate plutonium removal for each reagent or combination of reagents tested Screening tests showing greater than approximately 20 percent

plutonium removal were tested further using up to a maximum of six extraction stages These subsequent extraction stages are referred to as solvent extraction tests The specific technical approach performed for screening tests for soil samples #1 and #2 and the vegetation sample are described in the following sections

2.2.1 Soil Sample #1 Screening Tests

Fifteen screening tests were performed on soil sample #1 A summary of the test parameters used in the screening tests is provided in Table 2 1 Generally, 100 grams of soil were used for each test Reagents were added to the soil, as shown in Table 2 1, resulting in a liquid-to-solid ratio ranging from 1 1 to 100 1 by weight The liquid and soil mixture was then agitated for approximately 30 to 60 minutes at temperatures ranging from 34 degrees Fahrenheit (°F) to 190°F A small aliquot of extract solution was removed after each addition of reagent for gross alpha screening (discussed in Section 3 1 2) Screening tests showing greater than approximately 20 percent gross alpha removal were subjected to subsequent extraction (described in Section 2 3), up to a maximum of six extraction stages with modifications to the test parameters Test 1, which was a control test, used five extraction stages even though plutonium removal of less than 20 percent was anticipated

2.2.2 Soil Sample #2 Screening Tests

Eleven screening tests were performed on soil sample #2 A summary of the test parameters used in the screening tests is provided in Table 2 2 Generally, the test parameters were similar to those described in Section 2 2 1 For example, 100 grams of soil were used per test, reagents listed in Table 2 2 were added to the soil resulting in a liquid-to-solid ratio ranging from 1 1 to 8 1, the liquid and soil mixture was agitated for approximately 30 to 60 minutes at temperatures ranging from 34°F to 190°F, and a small aliquot of extract solution was removed after each addition of reagent for gross alpha screening Screening tests showing greater than approximately 20 percent gross alpha removal were subjected to subsequent extraction (described in Section 2 3) with modifications to the test parameters

2.2.3 Vegetation Sample Screening Tests

Eight screening tests were performed on the vegetation sample A summary of the test parameters used in the screening tests are provided in Table 2 3 Generally, the test parameters were similar to those described in Sections 2 2 1 and 2 2 2 For example, 50 grams of vegetation were used per test reagents listed in Table 2 3 were added to the vegetation resulting in a liquid-to-solid ratio of 8 1, the liquid and vegetation mixture was agitated for approximately 30 to 90 minutes at temperatures ranging from less than 34°F to 190°F and a small

aliquot of extract solution was removed after each addition of reagent for gross alpha screening Screening tests showing greater than approximately 20 percent gross alpha removal were subjected to subsequent extraction (described in Section 2 3), extraction with modifications to the test parameters

2.3 Solvent-Extraction Tests

Solvent-extraction tests were performed on each soil and vegetation screening test showing greater than approximately 20 percent gross alpha removal Each solvent-extraction test consisted of subsequent extraction stages following the screening test (the screening test being the first extraction stage of the solvent-extraction test) Generally, each extraction stage consisted of adding 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

2.3.1 Soil Sample #1 Solvent-Extraction Tests

Four screening tests (Tests 1, 4, 10, and 15) were subjected to subsequent solvent-extraction stages with modifications to the screening test parameters as shown in Table 2 4 These subsequent extraction stages will be referred to as solvent-extraction tests A step-by-step description of Test 15 is given below to provide further clarification of the extraction sequence used during Phase I sample testing

A 100-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 for 60 minutes at 150°F and allowed to settle The extract solution was sampled and analyzed using a gross alpha screening technique to obtain an estimate of the extraction efficiency of the peroxide solution

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 again agitated for 60 minutes at 160°F and allowed to settle The extract solution referred to as interstage extract solution, was analyzed using a gross alpha screening technique to obtain an estimate of the extraction efficiency of the peroxide/citric acid solution This extract solution free of suspended

solids, was sampled and later analyzed for isotopic plutonium The extracted solids referred to as interstage solids, were sampled and later analyzed for isotopic plutonium

The above extraction procedure was repeated three more times, starting with the addition of hydrogen peroxide, to the solids and liquid remaining in the extraction vessel, for a total of four extraction stages The final treated solids and final extract solution were later analyzed for total uranium and isotopic plutonium 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

2.3.2 Soil Sample #2 Solvent Extraction Tests

Four solvent extraction tests (Tests A, B, C, and D) were performed with modifications to the screening test parameters as shown in Table 2 5 The step-by-step procedure used for soil sample #2 was similar to that discussed in Section 2 3 1 for soil sample #1

2.3.3 Vegetation Sample Solvent Extraction Tests

Four solvent extraction tests (Tests V-1, V-2, V-3, and V-7) were performed with modifications to the screening test parameters as shown in Table 2 6 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 Screening 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	Further Testing
1	---	---	Triethylamine	1	60 min	<40°F	Y
2	---	NA_2CO_3	Triethylamine	1	30 min	Room temp	N
3	H_2O_2	NA_2CO_3	Triethylamine	1	30 min	<40°F	N
4	H_2O_2	$C_6H_8O_7$	Triethylamine	1	30 min	150°F *	Y
5	$Na_2S_2O_4$	$Na_3C_6H_5O_7$	Triethylamine	1	30 min	<40°F	N
6	HNO_3	$C_6H_8O_7$	Triethylamine	1	30 min	150°F *	N
7	HNO_3	---	---	1	30 min	Room temp	N
8	H_2O_2	$NA_2CO_3/Na_3C_6H_5O_7$	---	1	60 min	160°F	N
9	H_2O_2	---	Aliquot 336™	1	30 min	Room temp	N
10	$Na_2S_2O_4$	$Na_3C_6H_5O_7$	---	1	60 min	150°F	Y
11	$Na_2S_2O_4$	$C_{12}H_{27}O_4P$	Triethylamine	1	60 min	Room temp	N
12	---	$C_{12}H_{27}O_4P$	Triethylamine	1	30 min	<40°F	N
13	H_2O_2	$C_{12}H_{27}O_4P$	---	1	30 min	160°F	N
14	H_2O_2	$C_6H_8O_7/C_{12}H_{27}O_4P$	---	1	30 min	160°F	N
15	H_2O_2	$C_6H_8O_7$	---	1	60 min	160°F	Y

---	None
<	Less than
°F	Degrees Fahrenheit
$C_6H_8O_7$	Citric acid
$C_{12}H_{27}O_4P$	Tributyl phosphate
H_2O_2	Hydrogen peroxide
HNO_3	Nitric acid
min	Minute
N	No
$Na_3C_6H_5O_7$	Sodium citrate
NA_2CO_3	Sodium carbonate
$Na_2S_2O_4$	Sodium dithionite
temp	Temperature
Y	Yes

* Hot aqueous extractions followed by cold (<40°F) triethylamine extractions

Table 2 2. Summary of Screening 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	Further Testing
A	$Na_2S_2O_4$	$Na_3C_6H_5O_7$	---	1	60 min	160°F	Y
B	H_2O_2	$C_6H_8O_7$	---	1	60 min	190°F	Y
C	$Na_2S_2O_4$	$Na_3C_6H_5O_7$	---	1	60 min	190°F	Y
D	H_2O_2	$C_6H_8O_7$	Triethylamine	1	30 min	190°F	Y
E	HNO_3	---	Triethylamine	1	60 min	190°F	N
			Aliquot 336™				
F	HNO_3	$C_{12}H_{27}O_4P$	Triethylamine	1	60 min	190°F	N
G	HNO_3	$C_{12}H_{27}O_4P$	Aliquot 336™	1	60 min	Room temp	N
H	HNO_3	---	Sodium Nitrite	1	60 min	Room temp	N
			Aliquot 336™				
J	$Na_2S_2O_4$	---	Hydrochloric	1	60 min	190°F	N
			Acid, Sodium				
			Chloride				
K	H_2SO_4	---	Sodium Sulfate	1	60 min	190°F	N
			Sulfate				
L	---	---	Triethylamine	1	30 min	<40°F	N

---	None
°F	Degrees Fahrenheit
$C_6H_8O_7$	Citric acid
$C_{12}H_{27}O_4P$	Tributyl phosphate
HNO_3	Nitric acid
H_2O_2	Hydrogen peroxide
min	Minute
N	No
$Na_3C_6H_5O_7$	Sodium citrate
$Na_2S_2O_4$	Sodium dithionite
temp	Temperature
Y	Yes

Table 2.3. Summary of Screening 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	Further Testing
V-1	H_2O_2	$C_6H_8O_7$	Triethylamine	1	60 min	190°F	Y
V-2	$Na_2S_2O_4$	$Na_3C_6H_5O_7$	Triethylamine	1	60 min	190°F	Y
V-3	H_2O_2	$C_6H_8O_7$	Triethylamine	1	60 min	<40°F	Y
V-4	H_2O_2	$Ca(NO_3)_2$	---	1	60 min	Room temp	N
V-5	---	$Ca(NO_3)_2$	---	1	90 min	Room temp	N
V-6	---	$Ca(NO_3)_2$	---	1	90 min	Room temp	N
V-7	NaOCl	---	---	1	30 min	170°F	Y
V-8	HNO_3	$C_6H_8O_6$	---	1	30 min	Room temp	N

---	None
<	Less than
°F	Degrees Fahrenheit
$C_6H_8O_6$	Ascorbic acid
$C_6H_8O_7$	Citric acid
$Ca(NO_3)_2$	Calcium nitrite
H_2O_2	Hydrogen peroxide
HNO_3	Nitric acid
min	Minutes
N	No
$Na_3C_6H_5O_7$	Sodium citrate
NaOCl	Sodium hypochlorite
$Na_2S_2O_4$	Sodium dithionite
temp	Temperature
Y	Yes

Table 2.4 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	---	---	Triethylamine	3	60 min	Ext #1,2 - <40°F Ext #3-5 - 100°F
4	H_2O_2	$C_6H_8O_7$	Triethylamine	3	30 min	150°F *
10	$Na_2S_2O_4$	$Na_3C_6H_5O_7$	---	4	60 min	150°F
15	H_2O_2	$C_6H_8O_7$	---	4	60 min	160°F

---	None
<	Less than
°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

* Hot aqueous extraction followed by cold (<40°F) triethylamine extraction

Table 2.5 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
A	$Na_2S_2O_4$	$Na_3C_6H_5O_7$	---	4	60 min	160°F
B	H_2O_2	$C_6H_8O_7$	---	4	60 min	190°F
C	$Na_2S_2O_4$	$Na_3C_6H_5O_7$	---	4	60 min	190°F
D	H_2O_2	$C_6H_8O_7$	Triethylamine	4	30 min	190°F

---	None
°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 2.6. 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
V-1	H_2O_2	$C_6H_8O_7$	Triethylamine	6	60 min	190°F
V-2	$Na_2S_2O_4$	$Na_3C_6H_5O_7$	Triethylamine	6	60 min	190°F
V-3	H_2O_2	$C_6H_8O_7$	Triethylamine	3	60 min	Ext 1,2 - <40°F Ext 3 - 190°F
V-7	NaOCl	---	---	2	30 min	170°F

---	None
<	Less than
°F	Degrees Fahrenheit
$C_6H_8O_7$	Citric acid
H_2O_2	Hydrogen peroxide
min	Minutes
$Na_3C_6H_5O_7$	Sodium citrate
NaOCl	Sodium hypochlorite
$Na_2S_2O_4$	Sodium dithionite

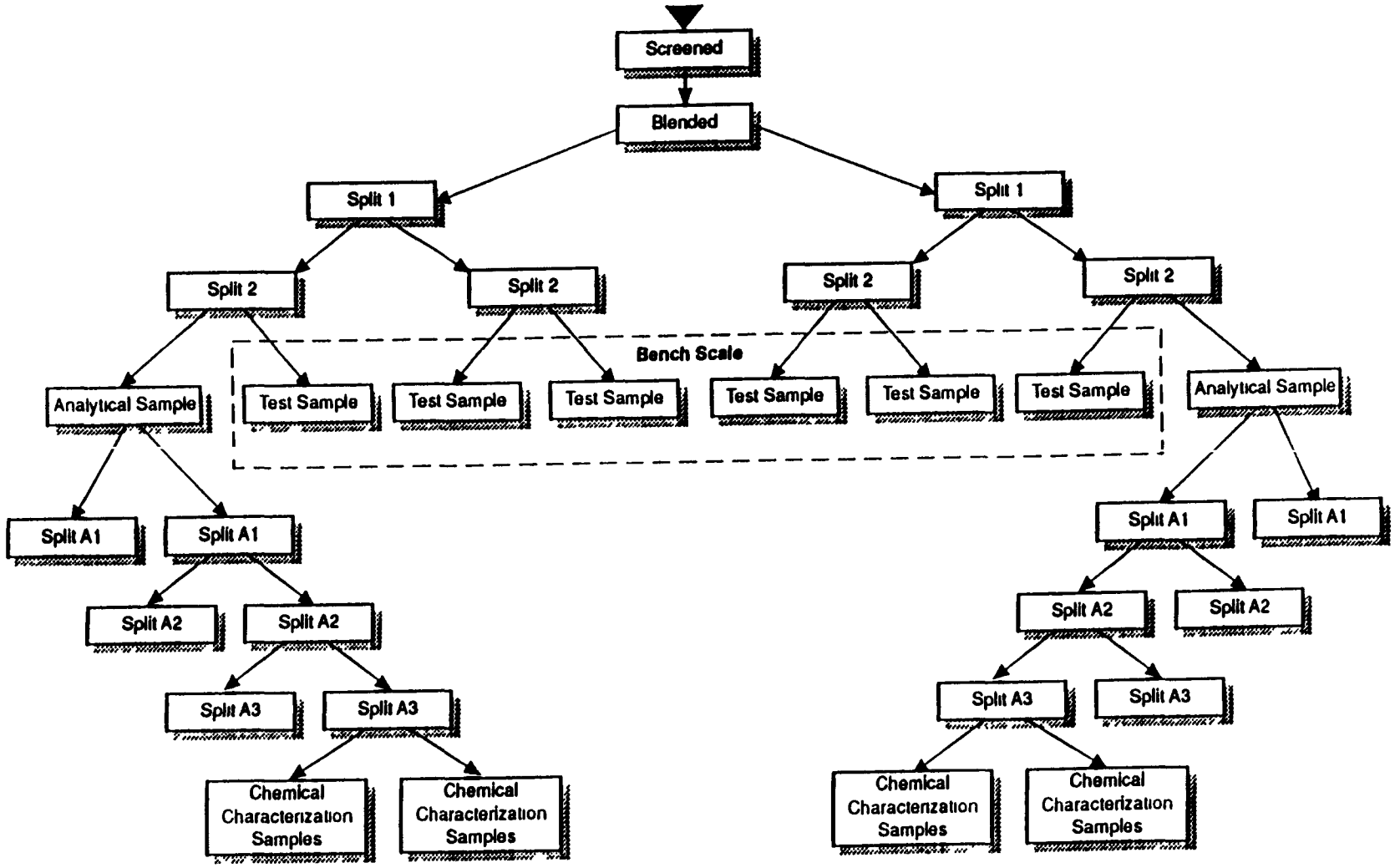


Figure 2 1

Soil Samples 1 and 2 Bench-scale Test and Analytical Sample Preparation

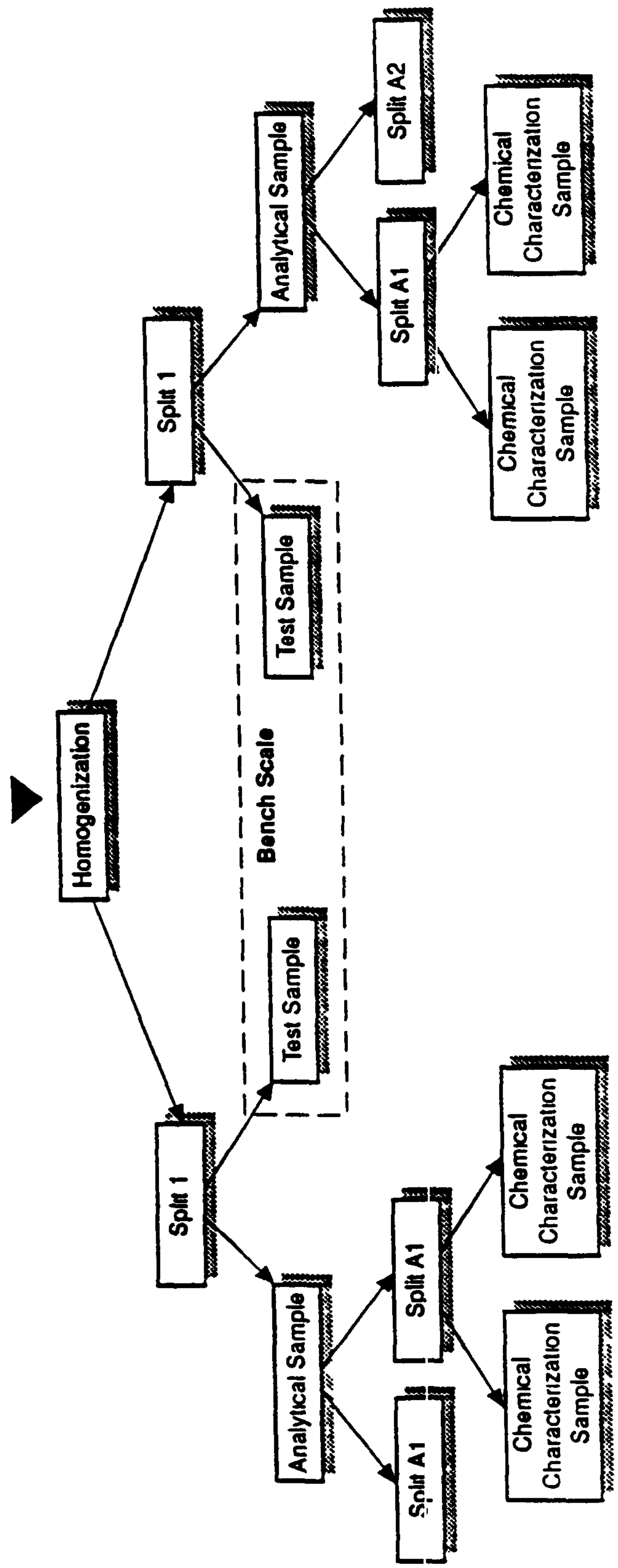
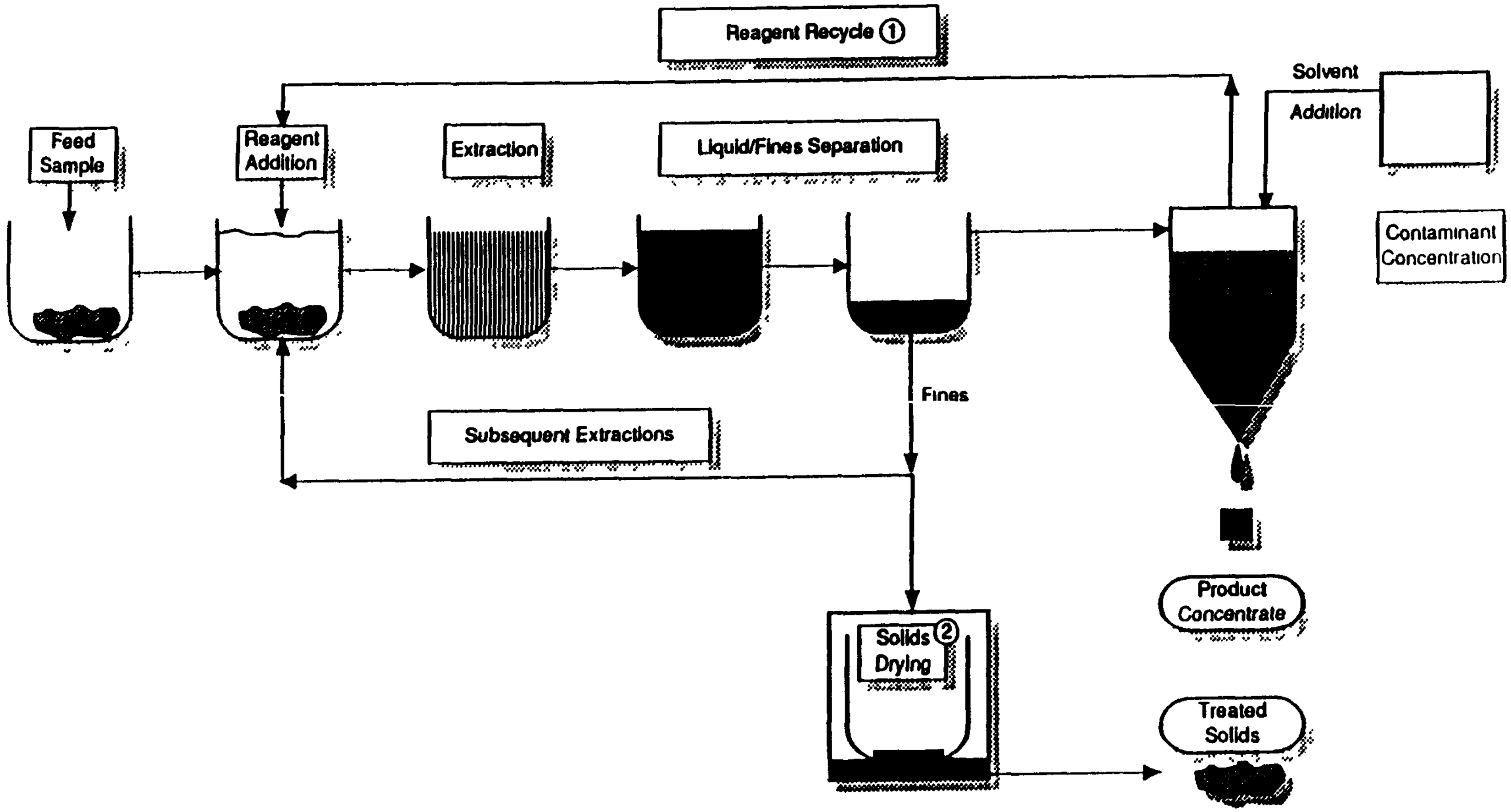


Figure 2 2

Vegetation Bench-scale Test and Analytical Sample Preparation



Explanation

① Reagent recycle only performed after the completion of all extraction stages

② Solids drying only performed after the completion fo all extraction stages

Figure 2 3

Bench-scale Solvent Extraction Process for Removal of Radionuclides

3 0 ANALYTICAL AND QUALITY ASSURANCE/QUALITY CONTROL PROGRAM DESIGN

As described in Section 2 0, the sampling and analytical program was conducted by the bench-test technician in support of the Phase I 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 reported analytical results The analytical and QA/QC programs conducted for the Phase I 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 I bench-scale testing included three general components (1) chemical characterization of the RFP feed soil and vegetation samples prior to testing, (2) radioactivity screening analyses in support of Phase I bench-scale screening tests, and (3) radiochemical analyses for COCs in support of Phase I bench-scale solvent extraction tests Sample analyses for all three components of the Phase I 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 to be analyzed for the following radiochemical parameters Pu-238, Pu-239,240, 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 for the Phase I testing and were used to assess removals of COCs achieved by the Phase I tests

In addition to radiochemical analyses, the analytical sample splits from the feed samples underwent screening analyses for total solids oil and grease, buffering capacity (titration curve), and pH These analyses were performed on location by bench-test technicians using their internal SOPs Results from these analyses assisted in the design of the Phase I bench-scale tests

3 1 2 Radioactivity Screening Analyses for Bench-scale Screening Tests

As described in Section 2 2, the Phase I bench-scale testing program included screening tests using specific extraction formulations for effectiveness in removing COCs from the RFP feed soil and vegetation samples These screening tests generally involved the performance of a single extraction To estimate the effectiveness of the extraction in removing COCs, the extract solution generated from each screening test was submitted to the on-site laboratory to undergo semi-quantitative screening analyses for gross alpha activity Performance of these screening analyses involved modifying the on-site laboratory's SOP for gross alpha analysis to utilize a smaller sample aliquot (~1 ml), a simplified sample preparation procedure (i e , direct loading of sample onto a planchette), and an abbreviated counting time Although the screening analyses produced data with greater associated uncertainty than the on-site laboratory's standard method for gross alpha analysis, the screening analyses data allowed specific extraction approaches to be evaluated on an expedited basis during Phase I

3 1 3 Radiochemical Analyses for Bench-scale Solvent Extraction Tests

Extraction formulations and approaches showing promise based on screening results were more thoroughly investigated by performing additional extraction stages 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 I 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 I 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

Details of the QA/QC program for the solvent extraction bench-scale treatability study are provided in the QAA Data quality issues identified from the application of QA/QC protocols in the Phase I 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 I 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 each intermediate extraction stage	2 to 4[b]	x	x	
Interstage extract solution	After each intermediate extraction stage	2 to 4[b]	x	x	
Final treated solids	After final extraction stage	1	x	x	x
Final extract solution	After final extraction stage	1	x	x	x
Final extract concentrate (heavy phase)	After combining and concentrating all extract solutions from test	0 to 1[c]	x	x	x

Pu Plutonium
U Uranium

a. Includes major process media sampled during Phase I tests
b The total number of extractions performed varied between the different bench-scale tests (see Section 2 3)
c Not collected during all Phase I bench-scale tests

4 0 RESULTS AND DISCUSSION

This section presents the results of the Phase I 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 I Bench-scale Testing

The Phase I bench-scale tests produced both process and analytical results Process test results for each soil and vegetation sample included approximate 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 three components of the bench-scale testing sample preparation, screening tests, 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 The observed variations in the feed soil concentrations can be attributed to the analytical variance, based on the fact that all feed soil concentrations fell within the 90% confidence interval The variations in the vegetation feed concentrations may be attributed to analytical variance and the inherent heterogeneity of the sample matrix A statistical evaluation of the chemical characterization analytical results was performed and is discussed in Section 4 2 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

4 1 2 Soil Sample #1 Test Results

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

The extraction temperatures varied from 34 degrees Fahrenheit (°F) to 190°F

The extraction times varied from 30 minutes to approximately 14 hours

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 4

The ratio of feed to reagent (i e , oxidation/reduction and complexing agents), expressed on a weight-to-weight basis varied from 1 1 to 1 100

The ratio of solvent to reagent, expressed on a weight-to-weight basis, varied from 1 0 to 10 1

The results of Pu-239 240 and total uranium analyses conducted on soil sample #1 feed, interstage (i e , first extraction second extraction, etc), and final treated solids from Phase I solvent extraction tests are presented in Table 4 2 The analytical results show that in tests 10 and 15, plutonium is reduced from a mean feed concentration of 740 pCi/g to 86 pCi/g and 97 pCi/g, respectively, in the final treated solids Additional analytical results (i e , extract solution, extract concentrate) from Phase I, 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 I bench-scale testing of soil sample #2 are summarized as follows

The extraction temperatures varied from 34°F to 190°F

The extraction times varied from 30 to 60 minutes

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 5

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

The ratio of solvent to reagent, expressed on a weight-to-weight basis, was varied from 1 0 to 19 1

The results of Pu-239,240 and total uranium analyses conducted on soil sample #2 feed, interstage (i e , first extraction, second extraction, etc), and final treated solids from Phase I solvent extraction tests are presented in Table 4 3 The analytical results show that in tests A and C, Pu-239,240 is reduced from a mean feed concentration of 1350 pCi/g to 170 pCi/g and 180 pCi/g, respectively, in the final treated solids Additional analytical results (i e , extraction solution, extraction concentrate) from Phase I, soil sample #2 solvent extraction tests are presented in Appendix A

4 1.4 Vegetation Sample Test Results

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

The extraction temperatures varied from 34°F to 190°F

The extraction times varied from 30 to 90 minutes

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 6

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

The ratio of solvent to reagent, expressed on a weight-to-weight basis was 5 1

The results of Pu-239,240 and total uranium analyses conducted on feed, interstage (i e , first extraction, second extraction, etc), and final treated solids from Phase I solvent extraction vegetation testing are presented in Table 4 4 The analytical results show that in test V-2, Pu-239,240 is reduced from a mean feed concentration of 620 pCi/g to 87 pCi/g in the final treated solids The observed variability in the interstage solid results may reflect heterogeneities between the small sample aliquots used Additional analytical results (i e , extraction solution, extraction concentrate) from Phase I, vegetation sample solvent extraction tests are presented in Appendix A

4.2 EVALUATION OF PHASE I ANALYTICAL RESULTS

Data evaluation of Phase I analytical results was performed to assist in assessing the overall bench-scale test performance during Phase I testing The following three specific evaluations of Phase I analytical data were performed (1) a statistical evaluation of feed soil analytical results, (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 These statistics were calculated for Pu-239,240 These statistics were calculated 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 I 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 5 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

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 I 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

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

The percentages of Pu-239,240 removed for Phase I testing of soil sample #1 are summarized in Table 4 6 The results, based on the mean feed concentration, ranged from 55 to 88 percent removal of Pu-239,240 from soil sample #1 Test 10 demonstrated the maximum mean contaminant removal of 88 percent However results of tests 4, 10, and 15 were equivalent within analytical variance 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 I testing of soil sample #2 are presented in Table 4 7 The results, based on the mean feed concentration, ranged from 63 to 87 percent removal of Pu-239,240 from soil sample #2 Test A demonstrated the maximum mean contaminant removal of 87 percent However, results of tests A, B, and C were equivalent within analytical variance 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 I testing of the vegetation sample are presented in Table 4 8 The results, based on the mean feed concentration, ranged from 50 to 86 percent removal of Pu-239,240 from the sample Test V-2 demonstrated the maximum mean contaminant removal was 86 percent The parameters used during each of these tests 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 I 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 I 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

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

where

$\text{Pu-239,240 Mass}_{(in)}$	=	$\text{Conc}_{(feed)}$ x Mass of feed sample in grams (as received basis)
$\text{Conc}_{(feed)}$	=	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 9 Solids mass balance results for soil sample #1 ranged from 95 to 108 percent recovery Contaminant mass balance results for soil sample #1 show that the Pu-239,240 recoveries ranged from 47 to 107 percent With the exception of test 1, the Pu-239,240 recoveries during Phase I testing of soil #1 were within the laboratory's control limits of 75 to 125 percent. Recoveries outside of this range may be caused by matrix effects, sample inhomogeneity, and/or variations in sample preparation protocols

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 10 Solids mass balance results for soil sample #2 ranged from 87 to 96 percent recovery Contaminant mass balance results for soil sample #2 show that the Pu-239,240 recoveries ranged from 62 to 98 percent With the exception of test B, the Pu-239,240 recoveries were within the laboratory's control limits of 75 to 125 percent for blank spike recoveries

4.2.3 3 Vegetation Sample Solids and Contaminant Mass Balance Results

Solids and Pu-239,240 mass balance results for the vegetation sample are summarized in Table 4 11 Solids mass balance results for the vegetation sample ranged from 63 to 105 percent recovery Contaminant mass balance results for the vegetation sample show that the Pu-239,240 recoveries ranged from 114 to 134 percent These recoveries are near or within the laboratory's control limit range of 80 to 120 percent recovery for blank spike samples In addition, recoveries of greater than 100 percent for the vegetation sample may be attributed to variability in the total solids analysis of the feed sample

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

pCi/g Picocuries per gram
NA Not analyzed

Table 4 2 Summary of Soil Sample #1 Solids Analytical Results

	Results	
	Pu-239,240 (pCi/g)	**Total U (µg/g)**
Test 1		
Feed*	740	12
5th ext (final treated) Solids	340	5 4
Test 4		
Feed*	740	12
1st ext. Solids	320	2 7
2nd ext Solids	190	1 7
3rd est (final treated) Solids	1 30	1 0
Test 10		
Feed*	740	12
1st ext Solids	200	#
2nd ext Solids	95	#
4th ext (final treated) Solids	86	1 4
Test 15		
Feed*	740	12
1st ext. Solids	120	#
2nd ext Solids	6 2	#
3rd ext. Solids	50	#
4th ext (final treated) Solids	97	0 86

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

* Mean feed concentration (discussed in Section 4 2 1)
\# Sample not analyzed for total uranium

Table 4 3 Summary of Soil Sample #2 Solids Analytical Results

	Results	
	Pu-239,240 (pCi/g)	**Total U (µg/g)**
Test A		
Feed*	1350	6 2
1st ext Solids	350	#
2nd ext Solids	220	#
3rd ext Solids	180	#
4th ext (final treated) Solids	170	1 4
Test B		
Feed*	1350	6 2
1st ext Solids	490	#
2nd ext Solids	400	#
3rd ext. Solids	230	#
4th ext. (final treated) Solids	230	0 90
Test C		
Feed*	1350	6 2
3rd ext Solids	170	#
4th ext. (final treated) Solids	180	#
Test D		
Feed*	1350	6 2
4th ext (final treated) Solids	500	1 3

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

* Mean feed concentration (discussed in Section 4 2 1)
\# Sample not analyzed for total uranium

Table 4 4 Summary of Vegetation Solids Analytical Results

	Results	
	Pu-239,240 (pCi/g)	Total U (µg/g)
Test V-1		
Feed*	620	5 7
3rd ext Solids	110	#
4th ext Solids	81	#
6th ext (final treated) Solids	220	0 70
Test V-2		
Feed*	620	5 7
3rd ext Solids	1 3	#
4th ext Solids	9 0	#
6th ext (final treated) Solids	8 1	0 70
Test V-7		
Feed*	620	5 7
1st ext. Solids	370	#
2nd ext (final treated) Solids	310	1 4

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

* Mean feed concentration (discussed in Section 4 2 1)
\# Sample not analyzed for total uranium

Table 4 5 Phase I 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	5	740	340	55
4	3	740	130	82
10	4	740	86	88
15	4	740	95	87

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
A	4	1,350	174	87
B	4	1,350	230	83
C	4	1,350	180	86
D	4	1,350	500	63

pCi/g Picocuries per gram

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

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
V-1	4	620	220	64
V-2	4	620	87	86
V-7	2	620	310	50

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	102	47
4	108	77
10	95	97
15	96	107

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)
A	96	98
B	87	62
C	94	79
D	93	84

* Based on mean feed concentrations

Table 4 11 Vegetation Sample Solids and Pu-239 240 Mass Balance Results

Test Identification	Solids Recovery (percent)	Pu-239,240* Recovery (percent)
V-1	63	127
V-2	105	134
V-7	82	114

* Based on mean feed concentrations

5.0 SUMMARY OF RESULTS AND RECOMMENDATIONS

This section summarizes the RFP soil and vegetation Phase I bench-scale test results and provides recommendations based on these results. Phase I bench-scale solvent extraction tests were evaluated to (1) generate performance data for removing plutonium from RFP soil and vegetation, (2) identify near optimum operating parameters (i.e., number of extraction stages, extraction temperature, pH, solvent ratios, and pretreatment requirements) for removing Pu-239,240 from RFP soil and vegetation, (3) calculate a Pu-239,240, and solids mass balance for each of the solvent extraction tests performed during Phase I, (4) calculate the percent of Pu-239,240 removed from RFP soil and vegetation after solvent extraction testing, and (5) evaluate the potential of Phase II bench-scale testing to remove Pu-239,240 from the soil and vegetation sample to concentrations at or below the treatability study benchmarks (TSBs).

The results of the Phase I 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 I testing. The Pu-239,240 removal for the tests with the most favorable results, following four extractions, was 88 percent for soil sample #1, 87 percent for soil sample #2, and 86 percent for the vegetation sample. Pu-239,240 and solids mass balance results, summarized in Table 5-1, support the Pu-239,240 removal results. Solids recovery ranged from 95 to 105 percent and Pu-239,240 recovery ranged from 97 to 134 percent.

Based on the results of the Phase I bench-scale solvent extraction testing, RCC's solvent extraction process is an effective treatment for the removal of Pu-239,240 from RFP soil and vegetation. Therefore, proceeding with Phase II testing is recommended to identify near optimum extraction parameters, allowing evaluation of the potential of solvent extraction to remove Pu-239,240 from the soil and vegetation samples to concentrations at or below the TSBs.

It is recommended that two tests of RFP soil be conducted during Phase II, one test using sodium dithionite and sodium citrate, and a second test using peroxide and citric acid. It is also recommended that one test of RFP vegetation be conducted during Phase II, using a combination of sodium dithionite and sodium citrate.

Table 5 1. Summary of Phase I Bench-scale Test Results

Sample	Number of Extractions	Pu-239,240 Removal (percent)	Pu-239,240 Recovery (percent)	Solids Recovery (percent)
Soil sample #1	4	88	97	95
Soil sample #2	4	87	98	96
Vegetation sample	4	86	134	105

Appendix A

Analytical Results

Analyses Results

Sample ID	Description	Pu-239/240, pCi/g	Pu-238, pCi/g	KPA U, ug/g	Gross a, pCi/g	Gross b, pCi/g	Am-241, pCi/g	O&G, mg/kg
Soil Sample Number One								
94-07-177								
-02	Left Tree Side Split	556 5	9 595	8 12				28000
-03	Left Tree Side Split	447 93	7 171	8 01	572 38	29 72		
-04	Left Tree Side Split						144	
redo of -03	Redo after Grinding	798 97	12 51					
94-07-178								
-02	Feed Sample for Quick Gross Alpha				660 49	36 48		
-03	Test 1 Treated Solids	335 79	5 667	5 354				
-04	Test 1 Organic Concentrate	6 9	< 0 132	0 068				
-05	Test 4, 2 extract soln	51 985	0 789					
-06	Test 4, 3 extract soln, heavy phase	235 27	3 835	3 614				
-07	Test 4, 3 solids	321 59	5 324	2 659				
-08	Test 4, 3 sump concentrate	1 075	0 016					
-09	Test 4, 5 extract soln	26 584	0 384					
-10	Test 4, 6 solids	188 87	3 198	1 662				
-11	Test 4, 6 extract soln, heavy phase	87 308	1 445					
-12	Test 4, 10 extract soln, heavy phase	34 282	0 599	0 181				
-13	Test 5, 2 extract soln	72 949	1 1					
-14	Test 5, 3 extract soln, heavy phase	146	2 37					
-15	Test 4, Treated solids	134	2 18	1 011				
94-07-179								
-02	Test 6, 2 extract soln	19 144	0 288					
-03	Test 6, treated solids	952 57	15 55	2 663				
-04	Test 10, 1 extract soln, ovnt mix	66 731	1 06					
-05	Test 10, 1 solids	205 4	3 694					
-06	Test 10, 2 extract soln	6 383	0 107					
-07	Test 10, 2 solids	94 788	1 198					
-08	Test 10, 4 extract soln	0 403	0 006					
-09	Test 10, Treated solids	86 4	1 63	1 371				
-10	Test 10, 3 extract soln	0 848	0 017					
-11	Test 10, final heavy phase concentrate	84 4	1 37					
-14	Test 15, 1 solids	116 55	1 977					
-15	Test 15, 2 extract soln	11 944	0 186					
-16	Test 15, 2 solids	6 167	< 0 405					
-17	Test 15, 3 extract soln	2 018	0 022					
-18	Test 15, 3 solids	50 126	0 784					
-19	Test 15, 4 extract soln	1 358	< 0 034					
-20	Test 15, Treated solids	94 7	1 72	0 86				
-21	Test 15, concentrated extract soln	283	4 52					
-23	Test 15, 1 extract soln	68 7	1 06					
94-07-184								
-02	Right side tree split	526 41	8 829	8 34				
-03	Right side tree split	501 65	7 018	8 265				
Redo -02	redo	664 01	10 414					
Redo -03	redo	748 76	12 955					

Sample ID	Description	Pu-239/240, pCi/g	Pu-238, pCi/g	KPA U, ug/g	Gross a, pCi/g	Gross b, pCi/g	Am-241, pCi/g	O&G, mg/kg
Soil Sample Number Two								
94-08-049								
-02	Left side tree split	1530 2	23 865	6 634	1127 5	51 13		1200
-03	left side tree split	1514 3	24 27	6 435				
94-08-050								
-02	Test A, 1 solids	353 09	5 914					
-03	Test A, 1 extract soln	109 73	1 654					
-04	Test A, 2 solids	219 29	4 284					
-05	Test A, 2 extract soln	15 806	0 225					
-06	Test A, 3 solids	182 73	3 414					
-07	Test A, 3 extract soln	4 741	0 078					
-08	Test A, Treated solids	174	3 35	1 415				
-09	Test A, 4 extract soln	1 38	0 023					
-10	Test B, 1 extract soln	35 9	0 6					
-11	Test B, 1 solids	486 59	7 401					
-12	Test B, 2 extract soln	22 465	0 332					
-13	Test B, 2 solids	406 59	6 995					
-14	Test B, 3 extract soln	5 558	0 077					
-15	Test B, 3 solids	227 84	4 306					
-16	Test B, 4 extract soln	4 223	0 073					
-17	Test B, Treated solids	229	4 17	0 895				
-19	Test C, 3 solids	172 14	3 108					
-20	Test C, Treated solids	184	3 39	1 069				
-21	Test C, composite extract soln	29 762	0 467					
-22	Test D, 1st composite extract soln	0 034	< 0 085					
-23	Test D, 2nd composite extract soln	56 8	0 91					
-24	Test D, treated solids	496	7 59	1 269				
94-08-056								
-02	Right side tree split	1200 1	19 919	7 195				
-03	Right side tree split	1152 5	19 072	6 52			251 86	

Sample ID	Description	Pu-239/240, pCi/g	Pu-238, pCi/g	KPA U, ug/g	Gross a, pCi/g	Gross b, pCi/g	Am-241, pCi/g	O&G, mg/kg
Vegetation Sample								
94-08-296								
-01	Homogenized Feed						74 929	
-02	Left side tree split	680 78	10 766	5 935				
-03	Left side tree split	598 8	9 432	5 82				
94-08-297								
-02	Test V1, 1 extract soln	21 2	0 32					
-03	Test V1, 2 extract soln	7 76	0 1					
-04	Test V1, 3 extract soln	3 26	0 064					
-05	Test V1, 4 extract soln	1 06	< 0 033					
-06	Test V1, 6 extract soln	< 0 021	< 0 057					
-07	Test V1, 3 solids	109 36	< 2 049					
-08	Test V1, 4 solids	81 036	1 288					
-09	Test V1, Treated solids	221	3 58	0 695				
-10	Test V2, 1 extract soln	31 5	0 52					
-11	Test V2, 2 extract soln	5 67	0 078					
-12	Test V2, 3 extract soln	1 35	< 0 035					
-13	Test V2, 4 extract soln	0 25	< 0 024					
-14	Test V2, 6 extract soln	< 0 023	< 0 052					
-15	Test V2, 3 solids	13 162	< 0 887					
-16	Test V2, 4 solids	8 991	< 1 091					
-17	Test V2, Treated solids	87 2	1 68	0 875				
94-08-298								
-02	Test V7, 1 solids	366 05	5 865					
-03	Test V7, 1 extract soln	16 3	0 261					
-04	Test V7, 2 extract soln	6	0 093					
-05	Test V7, Treated solids	311 89	5 09	1 434				
94-08-299								
-02	Right side tree split	462 78	7 261	5 59	719 72	27 47		
-03	Right side tree split	724 59	11 099	5 41				

Appendix B

Mass Balance Data

Test 1 Plutonium-239/240 Mass Balance

Item Description	Mass,g	Concentration, pCi/g	Net Activity, pCi
ACTIVITY INPUT			
Feed	900 72	740	666532 8
Total Input			666532 8
ACTIVITY OUTPUT			
Treated Solids	802 8	335 79	269572
Organic Concentrate	132 4	6 9	914
Samples	116 5	not anal	NA
Total Output			270486
TOTAL INPUT	666532 8		
TOTAL OUTPUT	270486		
ERCENT RECOVERY	**40 6%**	Percent Removal	54 6%

Test 4 Plutonium-239/240 Mass Balance

Item Description	Mass,g	Concentration, pCi/g	Net Activity, pCi
ACTIVITY INPUT			
Feed	89 9	740	66526
Total Input			66526
ACTIVITY OUTPUT			
2 Extract sol'n	41 8	51 985	2173
3 Heavy Phase	84 4	235 27	19857
3 Concentrate	158 6	1 075	170
3 Solids	5 2	321 59	1672
5 Extract sol'n	39 9	26 584	1061
6 Extract sol'n	107 2	87 308	9359
6 Solids	5 4	188 87	1020
10 Extract sol'n	155	34 282	5314
Treated Solids	78 3	134	10492
Total Output			51118
TOTAL INPUT	66526		
TOTAL OUTPUT	51118		
ERCENT RECOVERY	**76 8%**	Percent Removal	81 9%

Test 10 Plutonium-239/240 Mass Balance

Item Description	Mass,g	Concentration, pCi/g	Net Activity, pCi
ACTIVITY INPUT			
Feed	90	740	66600
Total Input			66600
ACTIVITY OUTPUT			
1 Extract sol'n	758	66 731	50582
1 Solids	8 3	205 4	1705
2 Extract sol'n	710 2	6 383	4533
2 Solids	3 5	94 788	332
3 Extract sol'n	683 8	0 848	580
4 Extract sol'n	693 3	0 403	279
Treated Solids	75 2	86 4	6497
Total Output			64508
TOTAL INPUT	66600		
TOTAL OUTPUT	64508		
ERCENT RECOVERY	**96 9%**	Percent Removal	88 3%

Test 15 Plutonium-239/240 Mass Balance

Item Description	Mass,g	Concentration, pCi/g	Net Activity, pCi
ACTIVITY INPUT			
Feed	90	740	66600
Total Input			66600
ACTIVITY OUTPUT			
1 Extract sol'n	756 5	68 7	51972
1 Solids	3 2	116 55	373
2 Extract sol'n	760 7	11 944	9086
2 Solids	3	6 167	19
3 Extract sol'n	707 1	2 018	1427
3 Solids	4 5	50 126	226
4 Extract sol'n	728	1 358	989
Treated Solids	75 8	94 7	7178
Total Output			71268
TOTAL INPUT	66600		
TOTAL OUTPUT	71268		
ERCENT RECOVERY	**107 0%**	Percent Removal	87 2%

Test A Plutonium-239/240 Mass Balance

Item Description	Mass,g	Concentration, pCi/g	Net Activity, pCi
ACTIVITY INPUT			
Feed	87 5	1350	118125
Total Input			118125
ACTIVITY OUTPUT			
1 Extract sol'n	758 9	109 73	83274
1 Solids	5 1	353 09	1801
2 Extract sol'n	781 6	15 806	12354
2 Solids	4 6	219 29	1009
3 Extract sol'n	705 3	4 741	3344
3 Solids	4 3	182 73	786
4 Extract sol'n	663 1	1 38	915
Treated Solids	72 1	174	12545
Total Output			116028
TOTAL INPUT	118125		
TOTAL OUTPUT	116028		
ERCENT RECOVERY	**98 2%**	Percent Removal	87 1%

Test B Plutonium-239/240 Mass Balance

Item Description	Mass,g	Concentration, pCi/g	Net Activity, pCi
ACTIVITY INPUT			
Feed	87 5	1350	118125
Total Input			118125
ACTIVITY OUTPUT			
1 Extract sol'n	734 1	39 745	29177
1 Solids	4 8	486 59	2336
2 Extract sol'n	753 9	22 465	16936
2 Solids	7 1	406 59	2887
3 Extract sol'n	710 3	5 558	3948
3 Solids	6 9	227 84	1572
4 Extract sol'n	635 1	4 223	2682
4 Fines	1 9	879 95	1672
Treated Solids	54 8	229	12549
Total Output			73759
TOTAL INPUT	118125		
TOTAL OUTPUT	73759		
ERCENT RECOVERY	**62.4%**	Percent Removal	83 0%

Test C Plutonium-239/240 Mass Balance

Item Description	Mass,g	Concentration, pCi/g	Net Activity, pCi
ACTIVITY INPUT			
Feed	87 5	1350	118125
Total Input			118125
ACTIVITY OUTPUT			
Composite Extract sol'n	2714 6	29 762	80792
1 & 2 Solids	14 2		0
3 Solids	6 3	172 14	1084
Treated Solids	64 2	184	11813
Total Output			93689
TOTAL INPUT	118125		
TOTAL OUTPUT	93689		
ERCENT RECOVERY	**79 3%**	Percent Removal	86 4%

Test D Plutonium-239/240 Mass Balance

Item Description	Mass,g	Concentration, pCi/g	Net Activity, pCi
ACTIVITY INPUT			
Feed	8 75	1350	11812.5
Total Input			11812.5
ACTIVITY OUTPUT			
Comp Extract sol'n 1	96 1	0 034	3
Comp Extract sol'n 2	100 4	56 8	5703
Treated Solids	8 4	496	4166
Total Output			9872
TOTAL INPUT	11812.5		
TOTAL OUTPUT	9872		
ERCENT RECOVERY	**83 6%**	Percent Removal	63 3%

Test V1 Plutonium-239/240 Mass Balance

Item Description	Mass,g	Concentration, pCi/g	Net Activity, pCi
ACTIVITY INPUT			
Feed	16 7	620	10354
Total Input			10354
ACTIVITY OUTPUT			
1 Extract sol'n	294 5	21 2	6243
2 Extract sol'n	396 5	7 76	3077
3 Extract sol'n	402 6	3 26	1312
3 Solids	0 5	109 36	55
4 Extract sol'n	385 2	1 06	408
4 Solids	1 3	81 036	105
5 Extract sol'n	217 6	NA	
6 Extract sol n	204 2	0	0
Treated Solids	8 8	221	1945
Total Output			13146
TOTAL INPUT	10354		
TOTAL OUTPUT	13146		
ERCENT RECOVERY	**127 0%**	Percent Removal	64 4%

Test V2 Plutonium-239/240 Mass Balance

Item Description	Mass,g	Concentration, pCi/g	Net Activity, pCi
ACTIVITY INPUT			
Feed	16 7	620	10354
Total Input			10354
ACTIVITY OUTPUT			
1 Extract sol'n	307 5	31 5	9686
2 Extract sol'n	387 1	5 67	2195
3 Extract sol'n	369 2	1 35	498
3 Solids	1 2	13 162	16
4 Extract sol'n	372.5	0 25	93
4 Solids	2	8 991	18
5 Extract sol'n	215 6	NA	
6 Extract sol'n	198 3	0	0
Treated Solids	15 5	87 2	1352
Total Output			13858
TOTAL INPUT	10354		
TOTAL OUTPUT	13858		
ERCENT RECOVERY	**133 8%**	Percent Rem oval	85 9%

Test V7 Plutonium-239/240 Mass Balance

Item Description	Mass,g	Concentration, pCi/g	Net Activity, pCi
ACTIVITY INPUT			
Feed	16 7	620	10354
Total Input			10354
ACTIVITY OUTPUT			
1 Extract sol'n	325 2	16 3	5301
1 Solids	1 6	366 05	586
2 Extract sol'n	356 9	6	2141
2 Solids	12 1	311 89	3774
Total Output			11802
TOTAL INPUT	10354		
TOTAL OUTPUT	11802		
ERCENT RECOVERY	**114 0%**	Percent Removal	49 7%

Test 1 Solids Mass Balance

Item Description	Mass,g	Total Solids, %	Net Solids, g
SOLIDS INPUT			
Feed	1000 8	90 0%	900 72
Total Input			900 72
SOLIDS OUTPUT			
2 Solids	76 4	100 0%	76
4 Solids	40 1	100 0%	40
Treated Solids	802 8	100 0%	803
Total Output			919

TOTAL INPUT	900 72
TOTAL OUTPUT	919
ERCENT RECOVERY	**102 1%**

Test 4 Solids Mass Balance

Item Description	Mass,g	Total Solids, %	Net Solids, g
SOLIDS INPUT			
Feed	100	90 0%	90
Total Input			90
SOLIDS OUTPUT			
1 Solids	5 2	100 0%	5
2 Solids	5 4	100 0%	5
Reagent Solids	7 86	100 0%	8
Treated Solids	78 3	100 0%	78
Total Output			97

TOTAL INPUT	90
TOTAL OUTPUT	97
ERCENT RECOVERY	**107 5%**

Test 10 Solids Mass Balance

Item Description	Mass,g	Total Solids, %	Net Solids, g
SOLIDS INPUT			
Feed	100	90 0%	90 0
Reagent Heel	35	4 3%	1 5
Total Input			91 5
SOLIDS OUTPUT			
1 Solids	8 3	100 0%	8
2 Solids	3 5	100 0%	4
Treated Solids	75 2	100 0%	75
Total Output			87
TOTAL INPUT	92		
TOTAL OUTPUT	87		
ERCENT RECOVERY	**95 1%**		

>>>>>>> **Reagent Heel**

0 1M Sodium Dithionite
FW = 174 11g/M
TS = 17 4g/l (0 0174g/ml)

0 1M Sodium Citrate
FW = 258 07g/M
TS = 25 81g/l (0 0258g/ml)

Test 15 Solids Mass Balance

Item Description	Mass,g	Total Solids, %	Net Solids, g
SOLIDS INPUT			
Feed	100	90 0%	90 0
Reagent Heel	23 7	1 2%	0 3
Total Input			90 3
SOLIDS OUTPUT			
1 Solids	3 2	100 0%	3
2 Solids	3	100 0%	3
3 Solids	4 5	100 0%	5
Treated Solids	75 8	100 0%	76
Total Output			87
TOTAL INPUT	90		
TOTAL OUTPUT	87		
ERCENT RECOVERY	**95 8%**		

>>>>>>> **Reagent Heel**

0 1M Citric Acid
Used 11 5g to 1 1 DIW
TS = 11 5g/l (0 0115g/ml)

Test A Solids Mass Balance

Item Description	Mass,g	Total Solids, %	Net Solids, g
SOLIDS INPUT			
Feed	100	87 5%	87 5
Reagent Heel	54 1	4 3%	2 3
Total Input			89 8
SOLIDS OUTPUT			
1 Solids	5 1	100 0%	5
2 Solids	4 6	100 0%	5
3 Solids	4 3	100 0%	4
Treated Solids	69 1	100 0%	69
Residual	3	100 0%	3
Total Output			86
TOTAL INPUT	90		
TOTAL OUTPUT	86		
ERCENT RECOVERY	**95 8%**		

Test B Solids Mass Balance

Item Description	Mass,g	Total Solids, %	Net Solids, g
SOLIDS INPUT			
Feed	100	87.5%	87 5
Reagent Heel	29	1 2%	0 3
Total Input			87 8
SOLIDS OUTPUT			
1 Solids	4 8	100 0%	5
2 Solids	7 1	100 0%	7
3 Solids	6 9	100 0%	7
Treated Solids	56 7	100 0%	57
Centrate Solids	635 1	0 2%	1
Total Output			77
TOTAL INPUT	88		
TOTAL OUTPUT	77		
ERCENT RECOVERY	**87 4%**		

Test C Solids Mass Balance

Item Description	Mass,g	Total Solids, %	Net Solids, g
SOLIDS INPUT			
Feed	100	87 5%	87 5
Reagent Heel	56 5	4 3%	2 4
Total Input			89 9
SOLIDS OUTPUT			
1 Solids	8	100 0%	8
2 Solids	6 2	100 0%	6
3 Solids	6 3	100 0%	6
Treated Solids	64 2	100 0%	64
Total Output			85
TOTAL INPUT	90		
TOTAL OUTPUT	85		
ERCENT RECOVERY	**94 2%**		

Test D Solids Mass Balance

Item Description	Mass,g	Total Solids, %	Net Solids, g
SOLIDS INPUT			
Feed	10	87 5%	8 8
Reagent Heel	27	1 2%	0 3
Total Input			9 1
SOLIDS OUTPUT			
Treated Solids	8 4	100 0%	8
Total Output			8
TOTAL INPUT	9		
TOTAL OUTPUT	8		
ERCENT RECOVERY	**92 7%**		

Test V1 Solids Mass Balance

Item Description	Mass,g	Total Solids, %	Net Solids, g
SOLIDS INPUT			
Feed	50	33 4%	16 7
Reagent Heel	15 5	1 2%	0 2
Total Input			16 9
SOLIDS OUTPUT			
3 Solids	0 5	100 0%	1
4 Solids	1 3	100 0%	1
Treated Solids	8 8	100 0%	9
Total Output			11
TOTAL INPUT	17		
TOTAL OUTPUT	11		
ERCENT RECOVERY	**62 8%**		

Test V2 Solids Mass Balance

Item Description	Mass,g	Total Solids, %	Net Solids, g
SOLIDS INPUT			
Feed	50	33 4%	16 7
Reagent Heel	27 1	4 3%	1 2
Total Input			17 9
SOLIDS OUTPUT			
3 Solids	1 2	100 0%	1
4 Solids	2	100 0%	2
Treated Solids	15 5	100 0%	16
Total Output			19
TOTAL INPUT	18		
TOTAL OUTPUT	19		
ERCENT RECOVERY	**104 6%**		

Test V7 Solids Mass Balance

Item Description	Mass,g	Total Solids %	Net Solids, g
SOLIDS INPUT			
Feed	50	33 4%	16 7
Total Input			16 7
SOLIDS OUTPUT			
1 Solids	1 6	100 0%	2
Treated Solids	12 1	100 0%	12
Total Output			14
TOTAL INPUT	17		
TOTAL OUTPUT	14		
ERCENT RECOVERY	**82 0%**		