6/4/97 Reagent Preparation TM

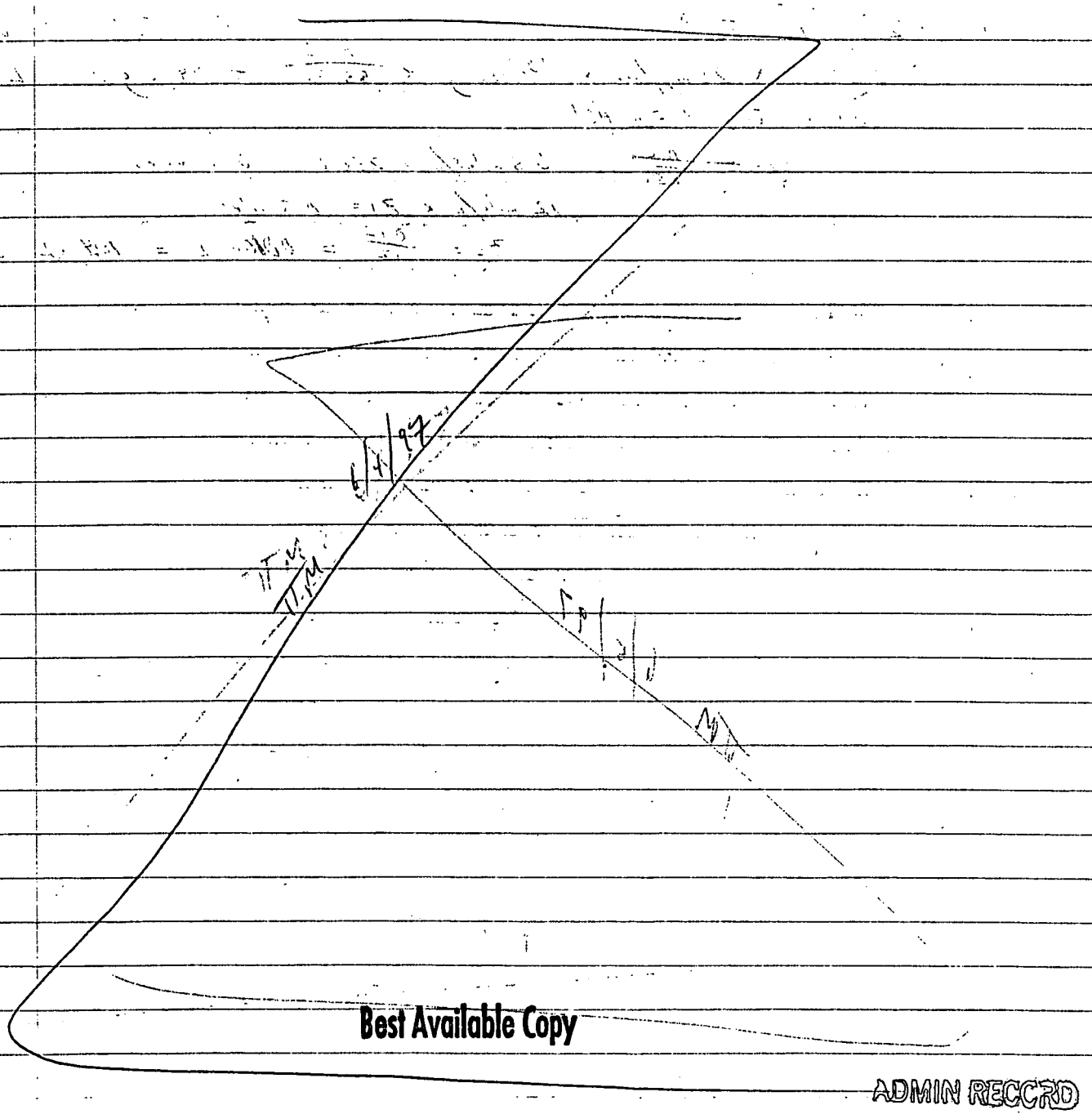
4:45 PM 1 M KNO_3 M.W. 101.1

1 mol/L x 0.5 L x 101.1 g/mol ≅ 50.6 grams / 500 mL

5:30 PM 1 M NaOAc M.W. ~~82.03~~ 136.08 as tri-hydrate TM

1 mol/L x 0.5 L x ~~82.03~~ 136.08 g/mol = ~~41.0~~ 68.0 grams / 500 mL 6/4/97

Adjusted to pH 5.0 w acetic acid



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ADMIN RECCRD

6/5/97 Reagent Preparation

11:05 AM 0.04 M hydroxylamine hydrochloride in 25% (v/v) HOAc

FW = 69.49

11:45 AM 0.04 moles/L × 1.0 L × 69.49 g/mole = 2.78 g/L

Added 250 mL glacial acetic acid and diluted to 1 liter with nanopure water.

12:15 20 mg/mL Fe^{+3} FW $FeCl_3 \cdot 6H_2O$ = 270.3

250 mL × 20 mg/mL × 1g/1000 mg × $\frac{270.3}{55.85}$ = 24.2 g/250 mL

dilute w/ 0.5 N HCl

$\frac{12\ N}{\times} = \frac{0.5\ N}{250}$ 0.5 moles/L × .250 L = 0.13 moles

12 mole/L × ? L = 0.13 moles

? L = $\frac{0.13}{12}$ = 0.0104 L = 10.4 mL conc. HCl

6/5/97

TM

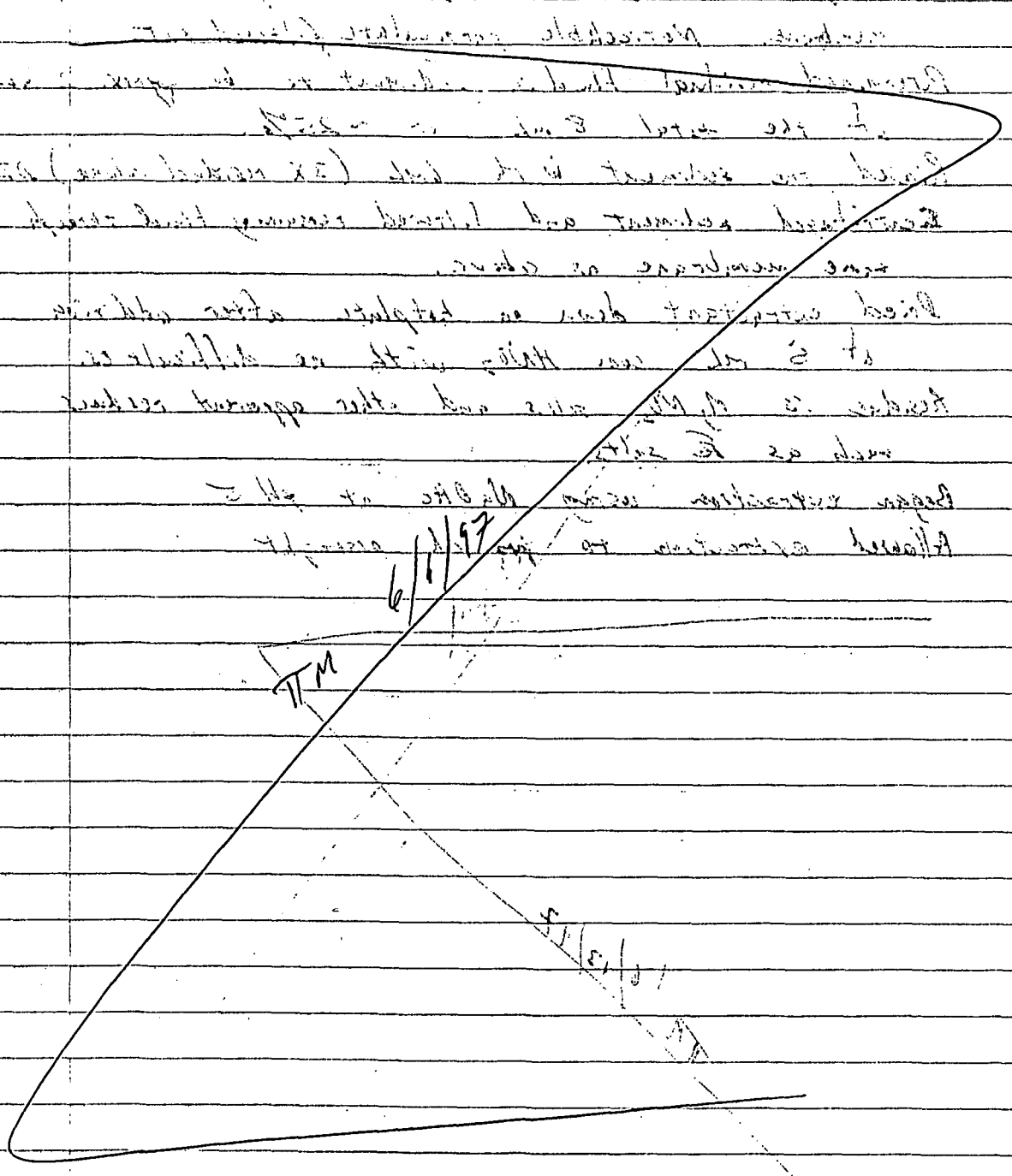
6/6/97 Reagent Prep

11:45 1 M $MgCl_2 \cdot 6H_2O$ FW 203.31

1 mol/L × ~~0.5 L~~ TM 6/6/97 1.0 L × 203.3 g/mol = 203.3 g

6/6/97

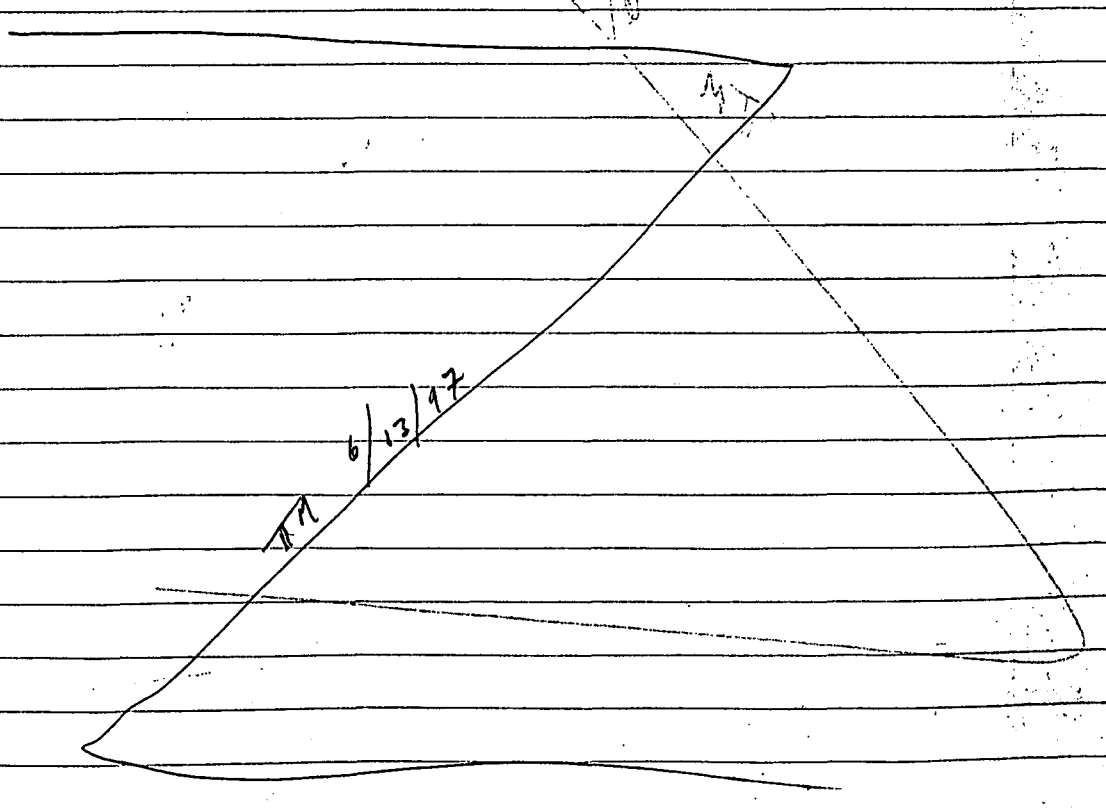
TM



6/12/97 Extraction series trial

Using sediment C2-02 3 grams
non-spiked solutions for trial of methid.
Actual weight - 3.01 g

Performed 1st extraction using $MgCl_2$
Centrifuged and decanted solution through 0.45 μ filter membrane. Noticeable particulate filtered out.
Determined residual fluid in sediment to be approx. 2 mL of the total 8 mL or ~25%.
Rinsed the sediment with 6mL (3X residual volume) DI H_2O
Centrifuged sediment and filtered remaining fluid through same membrane as above.
Dried extractant down on hotplate after addition of 5 mL conc. HNO_3 with no difficulties.
Residue is $Mg(NO_3)_2$ salts and other apparent residues such as Fe salts.
Began extraction using NaOAc at pH 5
Allowed extraction to proceed overnight.



6/13/97 Contamination of Trial sequential extraction

11:50 AM pH meter, Orion Model SA 720 S/N QX42A
calibrated between 4.00 & 7.00 using
Fisher Buffer Solution pH 4.00 Certified Lot # 946735-24
exp. date Oct. 1996 and
Fisher Buffer solution pH 7.00 certified Lot # 965245-24
exp. date Dec. 1998

12:00 PM Extractant from trial checked for pH after overnight extraction. Desired pH maintained 5.0 ± throughout extraction period. Check buffer solution verified pH to be 4.98 or 6.0, as desired.

6/13/97

VM

1/19/97

9:15AM Make-up of 0.02 M HNO_3.

.02 moles/L x 0.50 L = 1 moles/L x y L y = 0.625 mL

or ~~1 M x 0.5 L~~ 0.02 x .5 = 1 y y = 10 mL

Used: 10 mL 1M in 500 mL total, diluted with Nanopure H_2O at 18 megohms ~~TM~~

10:00AM Trial extraction. Organic extraction using 0.02 M HNO_3 and 30% H_2O_2. pH at 2.23 adjusted down with 1 M HNO_3. Only a few drops required. Began 2 h heating at 10:00 AM. Tube pressurized within a couple of minutes in the hot water bath. Must be vented to continue.

Previous extraction of oxides and hydroxides using hydroxylamine hydrochloride and acetic acid requires repeated treatments with HNO_3 and H_2O_2 to destroy the organic material, probably the acetate and amine components. Fe component requires a small amount of HCl to solubilize readily. Solution retains a cloudy appearance with translucent suspended material visible in the solution

1/19/97

1/25/97 Sample Transfer and preparation

4:05PM Samples 97L1879-001-006) were transferred from the original containers in which they were received to bottles. Samples 1-4 and sample 6 were received in sections wrapped with aluminum foil only. There was no custody seal on the samples and the integrity and containment was marginal. Upon inspection, there was no reason to believe that the structural profile of these samples had been maintained. It is this analysts understanding that samples were sectioned from the original lidded plastic containers in order to meet the non-radioactive shipping classification due to the specific activity found during the screening process. They ranged from 2 to 5 sections.

Sample 5 was maintained in the original sampling container and did have a custody seal.

For each sample the following protocol was followed:

1) Each fraction was thawed, unwrapped and combined with the other fractions of that sample.
2) The soil was blended together, clauds broken up, and large (> 0.5 cm) rocks removed. Roots and other organic debris were removed on the basis of its impossible ability to pass through a 0.5 cm sieve.
3) Sample 6 was left in a tub for 4 days to partially dry since it was too wet to dry sieve.
4) Moist Samples were place in 1 L wide mouth poly jars and labelled. Each sample was ~ 1 L.
5) ~ 200 - 250 mL of moist soil was sieved through a 2.36 mm sieve. The large debris was combined with the debris fraction. The passed material was put into 250 mL glass bottles and labelled.
6) a fraction (~ 10 grams) of the sieved soil was placed in a weighing dish and set into an

5/97 (cont.)

incubating oven at approx. 60°C overnight

- Ponds C-2 and B-5 samples were Fed-Exed to the Texas A & M facility. Samples were packed in styrofoam "peanuts" and were kept frozen using "dry" ice as the preservative.

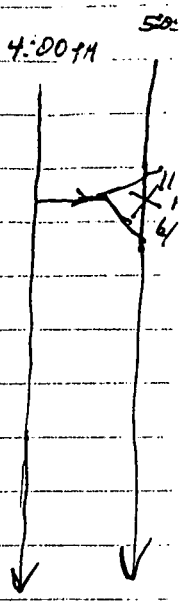
6/25/97

11 AM

6/26/97 Aliquot Samples

2:45 PM

Sample 97L1879 -	Mass (g)	1st Ext 1M	Time Beg.	End
001	3.05	$MgCl_2$	4:00 PM	5:00 PM
001D	3.07	"		
002	3.05	"		
002D	3.07	"		
003	2.99	"		
003D	2.99	KNO_3		
004	2.97	$MgCl_2$		
004D	3.04	KNO_3		
005	3.04	$MgCl_2$		
006	3.03	"		
B	3.06	"		



Sample 1D is a sample duplicate for precision measurement

Sample 2D is a duplicate of 2 whose extraction time has been lengthened.

Sample 3D and 4D are to compare with their duplicates 3 + 4 respectively with the use of KNO_3 as the original "exchangeable fraction" extraction.

All samples were aliquoted directly into 50 mL conical bottom plastic centrifuge tubes with sealing caps.

8 mL of the recorded extractant was added to each sample The samples were immediately capped and vortexed individually as an initial mixing process.

All samples were placed on their sides on top of a mixing (vibrating) plate.

D

6/27/97 Pipettor calibration

4:20 PM Cal of pipettor E-05

	DI water	Event	Mass	
	"	2	0.9911	
	"	3	0.9931	
	"	4	0.9913	
	"	5	0.9904	
		$\bar{x}$	0.9917	σ = 0.0010
			$\%2\sigma$ = 0.2	

Addition of Tracer 437.5284.32 Pu^{242} in 16% HNO_3

" 4.585 dpm/mL

" × (0.9917) mL

	vol added	activity added (dpm)
Sample 001	3	13.63
002	4	18.17
003	3	13.63
004	2	9.08
005	1	4.54
001D	3	13.63
002D	4	18.17
003D	3	13.63
004D	2	9.08
005D	1	4.54
006	1	4.54

10

7/1/97 Extraction of carbonates

3:00PM - Addition of NaOAc to samples @ 2:00 PM

- Begin agitation @ 2:05 PM

- Remove samples @ 7:45 PM

- Filter samples complete by 9:00 PM

- Sample 2D Removed following AM

7/2/97

11:55AM Samples rinsed with H_2O at varying quantities to rinse organic material off of filter membrane and filtering device. Samples refrigerated after vortexing and centrifugation.

Original solution dried down on hot plate overnight in the presence of ~2mL of conc. HNO_3. Rinsate added to beakers and placed on warm hot plate.

Addition of Pu-242 tracer:

Pipettor calibration using DI water pipettor # E-05

Event	weight
1	0.9929
2	0.9929
3	0.9937
4	0.9912
5	0.9851

$\bar{x} = 0.9912$ $\sigma = 0.0031 \approx 0.3\%$

Ex 2 – Carbonates

Sample #	x(0.9912 volume	Activity
001	2	9.08 dpm
1D	2	
002		
2D		
003		
3D		
004		
4D		
005		
006		
B		

7/2/97

TM

7/8/97 (Exchangeable) Extractable Fraction

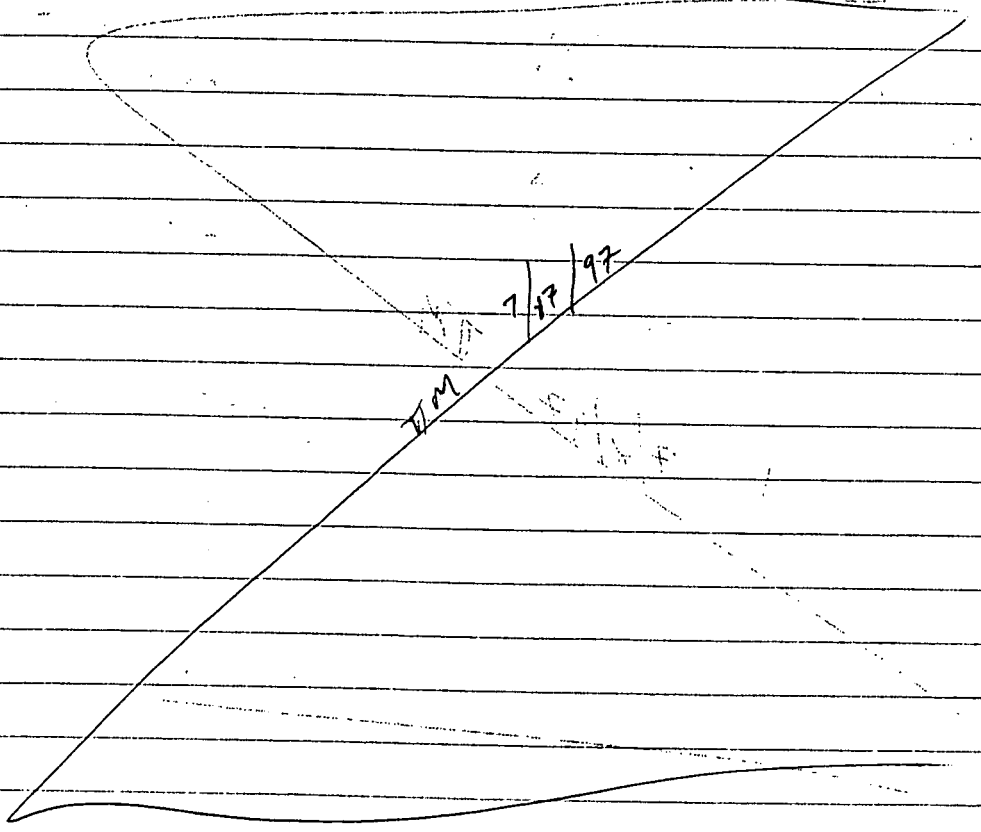
9:00 AM After addition of tracer samples were taken to dryness in the presence of conc. nitric acid and left on a low heat overnight. Samples were redissolved in dilute HNO_3, a small amount (~0.1 g) of $NaNO_2$ added to each and left to react for 20-30 minutes. Fe carrier was added (~20 mg) to each sample and the $Fe(OH)_3$ was precipitated using conc. NH_4OH. Samples were centrifuged and the precipitate redissolved in 3 times the volume of precipitate of 12 M HCl. Columns were prepared and samples were introduced to the columns on 7/9. It appeared that the quantity of Fe present was overwhelming the capacity of the columns, so the columns were stripped with dilute (~0.5 M) HCl and all solutions recombined and dried down on a hotplate.

TM 7/8/97

7/17/97 10:00 AM

Oxide and hydroxide extraction performed on 7/10/97 5:15 PM

Extraction began at 11:05 AM Completed at ~~4:15 PM~~ Except sample 20 which was extracted until 10:00 PM 7/10. All extracted as per Yong et al procedure As with other extractions. Extractant solvent was filtered through 0.45 μ 25 mm filter membrane. The residue on the filter (organic material + some sediment) was rinsed off of the filter into the sediment. ~~fr~~ Additional DI water was added to approximately 20 mL, vortexed to rinse out the solvent in the sediment, then refiltered through a 0.45μ 25mm membrane filter. This 2nd filter membrane was placed in the tube with the sediment for subsequent extraction rather than rinsing off the residue which would effectively dilute the subsequent extractant. Solvent was then dried down with a warm hot plate in 100 mL glass beakers.



7/17/97 Dilution of Tracer

3:40 PM 1.00 grams of 1695.6 dpm/g solution
NIST SRM 4334F

Dilute to 500 g total ~~mL~~ w 2M HNO_3

TM 7/17/97

1695.6 dpm / 500 g = 3.39 dpm/g

Dilute to 100 g w 2M HNO_3

1695.6 dpm / 100 g (TM) = 16.956 dpm/g

Actual: 1695.6 dpm / 100.02 g = 16.95 dpm/g

7/18/97 2:00 PM Spiking of Samples: Oxide w/ hydroxide frm

Sample	Spike Value	Mass of spike (g)	Activity (dpm)
1	16.95 dpm/g	0.5202	8.817
1D		0.5227	8.860
2		0.5211	8.833
2D		0.5206	8.824
3		0.5199	8.812
3D		0.5203	8.819
4		0.5192	8.800
4D		0.5160	8.746
5		0.5170	8.763
6		0.5180	8.780
B		0.5156	8.739

TM 7/18/97

7/18/96
4:30 PM
TM

Exchangeable fraction

7/23/97 pH adjustment of Organic extractant

11:45 AM

TM	Sample	Initial pH	Final pH	Water Bath Time In	Time
	97L1879-001	2.26	2.03	11:55 AM	1:0
	1D	2.35	2.01		
	2	2.34	2.02		
	2D	2.32	2.01		5:0
	3	2.77	2.00		1:05
	3D	2.82	2.01		
	4	2.88	2.01		
	4D	2.92	2.00		
	5	2.81	1.99		
	6	2.30	2.01		
	B	2.24	2.01		

All Samples were filtered through 0.45 μ membranes after centrifugation of the solids. The membrane was rinsed with the 3 ml of 30% H_2O_2 used for part II of the organics extraction protocol.

Part II Organic extraction

Sample	Initial pH	Final pH
001	2.89	2.03
1D	2.94	2.01
002	2.97	1.97
002D	~~3.43~~ TM 7/25/97	~~2.01~~ TM 7/25/97
003	3.43	2.01
3D	3.34	2.02
004	3.24	2.00
4D	3.24	2.00
005	3.23	1.97
006	2.96	1.97
B	2.67	1.99

7/28/97

TH 1:15 PM Part II pH adjustment

Sample	intial pH	final pH	Start digest	End digest
9712879-0020	2.79	1.99	1:20 PM	5:20

NH4 TH 7/28/97

1:50 PM Preparation of 3.2 M NaOAc in 20% v/v HNO_3

3.2 moles/liter X 77.08 g/mole X 0.25 L = 61.7 g in 250 mL

20% v/v HNO_3 acid = 50 mL 16 N HNO_3, 200 mL D.I. water

- Dissolve salts in 200 mL 20% v/v HNO_3
- Dilute to 250 mL total

2:30 PM Part III organic extraction

Residues are approximately 4 mL per tube

Added 5 mL 3.2 M NH4OAc in 20% v/v HNO_3

dilute to 24 mL line on tubes.

Samples	Extraction Start	Extraction End
0020	2:30 PM	3:00 PM
0010		
0030		
0040		
0050		
0060		
0080		
0020	10:5:41 PM	10:45 AM 7/29/97

TH 7/29/97

7/28/97 Spiking of Organic fraction

TM 4:45 PM

Sample	Spike Sp. Act.	mass of spike	Total Activity added (DPM)
97L1879-001	16.95 dpm/g	0.5230	8.86
001D		0.5199	8.81
002		0.5166	8.76
7/29/97 002D		~~0.5185~~ TM 7/28/97 0.5226	8.86
003		0.5185	8.79
003D		0.5196	8.81
004		0.5221	8.85
004D		0.5215	8.84
005		0.5216	8.84
006		0.5211	8.87
B		0.5226	8.81

7/29/97

7/30/97 Sample Count Data

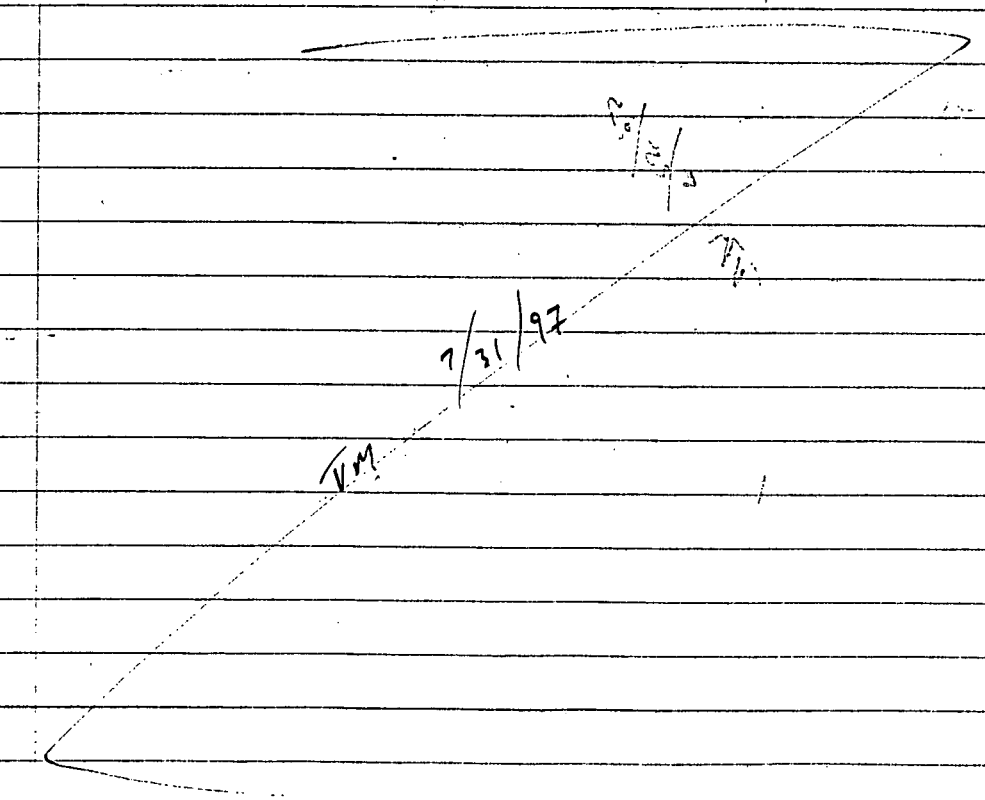
TM 9:00 PM

Sample	Fraction	(Integral) Tracer Counts	(Integral) Pu-239/240 Counts	Count Time (m)	Spiked Tracer DPM
971 1879-001	Exchang.	3472	232	1000	13.63
	Carbonate	1429	370	1000	9.08
	Sesquioxide	359	~~232~~ 1490 TM 7/30/97	150	8.82
	Organic	~~538~~ 284 (area)	20295	150	8.86
0010	Exchange	3745	246	1000	13.63
	Carbonate	2628	675	1000	9.08
	Sequioxide	348	1031	150	8.86
	Organic	~~517~~ 288 (area)	16807	154	8.81
002	Exchang.	3964	343	1000	18.17
	Carbonate	2618	1043	1000	9.08
	Sesquioxide	352	2677	150	8.83
	Organic	85 (area)	6403	30	8.76
0020	Exchange.	3577	~~2480~~	1000	18.17
	Carbonate	2756	838	1000	9.08
	Sesquioxide	387	1616	150	8.82
	Organic	146 (area)	9741	55	8.86
003	Exchang.	2867	40	1000	13.63
	Carbonate	2439	113	1000	9.08
	Sesquioxides	645	724	270	8.81
	Organic	429 (area)	20289	150	8.79
003D	Exchang.	3772	9	1000	13.63
	Carbonate	2567	112	1000	9.08
	Sesquioxides	641	649	270	8.82
	Organic	350 (area)	3995	154	8.81
004	Exchang.	2047	15	1000	9.08
	Carbonate	2502	62	1000	9.08
	Sesquioxide	648	537	270	8.80
	Organic	2385 (area)	11357	1000	8.85

7/30/97 TM

7/31/97 Sample Count Data (cont.)

7:20 AM	Sample	Fraction	(Integral) Tracer counts	(Integral) Pu-239/241 counts	count time (m)	
TM	9721879-0040	Exchang.	2687	1	1000	9
		Carbonate	2797	72	1000	9.
		Sesquioxide	617	468	270	8.
		Organic	2566	8468	1000	8.
	005	Exchang.	1135	4	1000	4.
		Carbonate	2621	18	1000	9.6
		Sesquioxide	1805	169	800	8.
		Organic	2379 (area)	2985	1000	8.
	006	Exchang.	1125	16	1000	4.
		Carbonate	2612	18	1000	9.
		Sesquioxide	2040	2536	800	8.
		Organic	1548 (area)	5001	640	8.
	Blank Soil	Exchang.	1028	1	1000	4.
		Carbonate	2033	1	1000	9.
		Sesquioxide	1659	4	800	8.
		Organic	2490	91	1000	8.



8/21/97 Sample Aliquots Dried & Sieved @ 2.36 mm

Sample	Wt (g)	
C2 #3	3.00	
C2 #5	3.00	
SID 25	3.03	
SID 27	3.02	
~~SID 39~~	~~3.00~~	Sample lost during filtration See next page for realiquot
Blank	3.02	

8/25/97 Extraction of Exchangeables

TM

Sample	wt (g)	Start ext	End
C2 #3	3.00	4:50	5:55
C2 #5	3.00		
SID 25	3.03		
SID 27	3.02		
SID 39	3.01	7:30	8:40
Blank	3.02	4:50	5:55

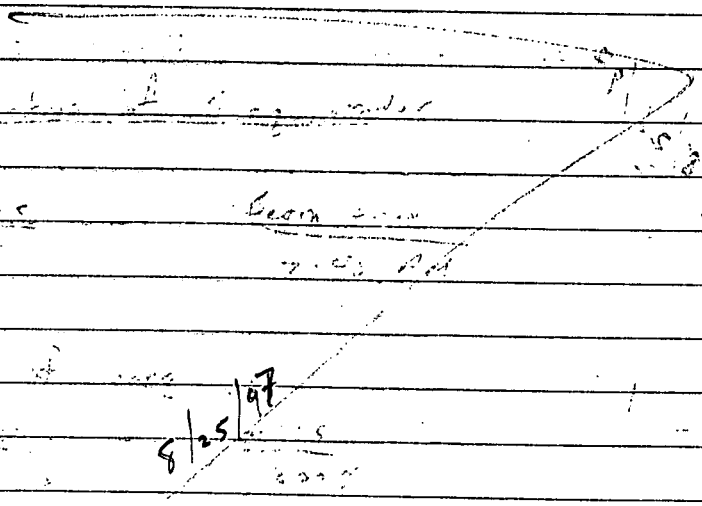
Tracer Addition 16.95 dpm/g → Pu-242

Sample	Vol of tracer (mL)	Activity	Pipetter Cal.
C2 #3	0.9874	16.74 dpm	0.9889
C2 #5			0.9907
SID 25			0.9850
SID 27			0.9871
SID 39			0.9851
Blank			

$\bar{x}$ = 0.9874

1σ = 0.0022

1σ % = 0.2%



8/25/97

8/26/97 pH Adjust for Carbonate Ext.

TM

Sample	Beginning pH	Final pH	Ext St	Ext End
c2 #3	5.09	4.99	6:50 AM	4:20 PM
c2 #5	5.56	4.99		
SID 25	5.06	5.00		
SID 27	4.97	4.97		
SID 31	5.39	4.98		
Blank	5.38	5.00		

8/26/97

TM

29

8/28/97 Tracer addition to Carbonate fxn

Pipette Cal.

Event	mass
1	0.9835
2	0.9851
3	0.9846
4	0.9826
5	0.9835
6	0.9793
7	0.9807
	$\bar{x}$ 0.9828

1σ = 0.0019 % unc. (1σ) = 0.2 %

Sample	mass	Activity
C2 #3	0.9828	16.7 dpm
C2 #5		
SED 25		
SED 27		
SED 39		
Blank 2		

Extraction of Sesquioxides

Samples	Begin time	End Time
All	7:40 PM	2:15 AM 8/29

Spike of Sesq.

Sample	mass	Act. Vity
C2 #3	0.9828	16.7 dpm
C2 #5		
SED 25		
SED 27		
SED 39		
Blank 2		

8/30/97 pH Adjustment for Organic Ext.

TM	Sample	Initial pH	Final pH	Start Ext.	End Ext.
	C2 #3	2.46	2.01	5:15 PM	7:34 PM
	C2 #5	2.67	2.03		
	SID 25	2.64	2.02		
	SID 27	2.93	2.00		
	SID 39	2.59	2.03		
	Blank	2.54	2.01		

8/31 II Part Org. Ext.

Sample	Initial pH	Final pH	St. Ext.	End Ext
C2 #3	2.59	1.99	1:05 PM	3:15 PM
C2 #5	2.62	2.00		
SID 25	2.49	2.01		
SID 27	2.45	2.01		
SID 39	2.49	1.99		
Blank	2.47	2.00		

Part III Ext. Start: 4:20 PM End: 5:20 PM

Spike of Org Samples

Sample	vol/mass	activity	Pipettor Cal. Event #	
			1	0.9
C2 #3	0.9582	16.8 dpm	2	0.98
C2 #5			3	0.98
SID 25			4	0.98
SID 27			5	0.98
SID 39			6	0.98
Blank			7	0.98
			$\bar{x}$ =	0.98
			σ =	0.00

9/14 Record of incubator temp at highest setting at hottest area
11:20 AM at system with hood closed (low air flow)
TM [60.0°C] This is the hottest temp that drying samples have been exposed to during this project.

9/25 Residual Fraction

Sample	Vessel #
97LR79-01	8
-02	7
-03	1
-04	4
-05	2
-06	5
Blank 1	3
C2 #3	9
~~C2 #5 STD 25~~ TM	6

9/25/97

~~Spike of Samples~~

C2 #1

9/25/97

9/25

	Sample	Fxn	Int Tracer Counts	Pu 239/240 Counts	Count Time	Spike (Tracer) DPM
TM	C2 #3-Ex		4861	7	1000	
9:19 PM		Carb	4033	33		
		Sesq	4847	77		
		Org	5520	25		
TM						
	C2 #5	Ex	4947	7	1000	
		Carb	4029	26		
		Sesq	5578	268		
		Org	5688	345		
	SID 25	Ex	4294	4	1000	
		Carb	3890	18		
		Sesq	5319	90		
		Org	5238	402		
	SID 27	Ex	4961	17	1000	
		Carb	~~4961~~ 4353 TM 9/25/97	~~17~~ 14 TM 9/25/97		
		Sesq	5591	~~14~~ 158		
		Org	5941	638		
					1000	
	SID 39	Ex	3062	8		
		Carb	3580	8		
		Sesq	5661	14		
		Org	5078	12		
	Blank 2	Ex	4264	10	1000	
		Carb	3676	14		
		Sesq	5579	18		
		Org	3889	11		

9/27 Pipettor Cal.
1:06 PM
TM

Event	Mass DI H_2O
1	0.9849
2	0.9894
3	0.9894
4	0.9866
5	0.9854
6	0.9857
7	0.9860

$\bar{x} = 0.9868$ $1\sigma = 0.0017$

9/27 Spike of Samples 16.95 dpm/g

Sample	Mass of Spike	Activity
01	0.9868	16.7 dpm
02		
03		
04		
05		
06		
C2 #3		
C2 #5		
~~SED 25~~		
Blank 1		
~~C2 #5~~		
~~SED 27~~		
~~SED 39~~		

TM 9/28/97

TM 9/27/97

9/28 Spike of Samples

TIM	Sample	Mass of Spike	Activity in Sample
1050AM	SID 25	0.9868	16.7 dpm
	SID 27		
	Blank 2		

9/28 Spectral Quantification

9:30

PM	Sample	Exn	Int Tracer Counts	Pu 239/240 counts	Count Time	Tracer DPM
	972 1879-01	Residual	1159 (area)	68818	500	16.7
	C2 #2		1123 (area)	120243	500	16.7
	C2 #3		27	147	500	16.7
	C2 #4		22	147	500	16.7
	C2 #5		1866	642	500	16.7
	C2 #6		2082 (area)	16889	500	16.7
	Blank 1		1574	83	500	16.7
	C2 #3		1984	329	500	16.7
	C2 #5		1979	1652	500	16.7
	SID 25		211	144	500	16.7
	SID 27		58	143	500	16.7
	Blank 2		105	2	500	16.7

10/15 Spike of Residual Fractions

TM

10/15/97

Spike Residual Fxns

10/15

Sample	Spike Activity	Mass	Total Activity (dpm)
1D	16.95 dpm/g	10 x 0.9892	167.7
2D		10 x	167.7
3D		2 x	33.53
4D		2 x	33.53
SID 39		2 x	33.53

Pipetter CAL

Event	Σmass
~~1~~	~~0.9828~~
2	0.9926
3	0.9903
4	0.9898
5	0.9903
6	0.9902
7	0.9867
8	0.9845

$\bar{x} = 0.9892 / s = 0.0027$

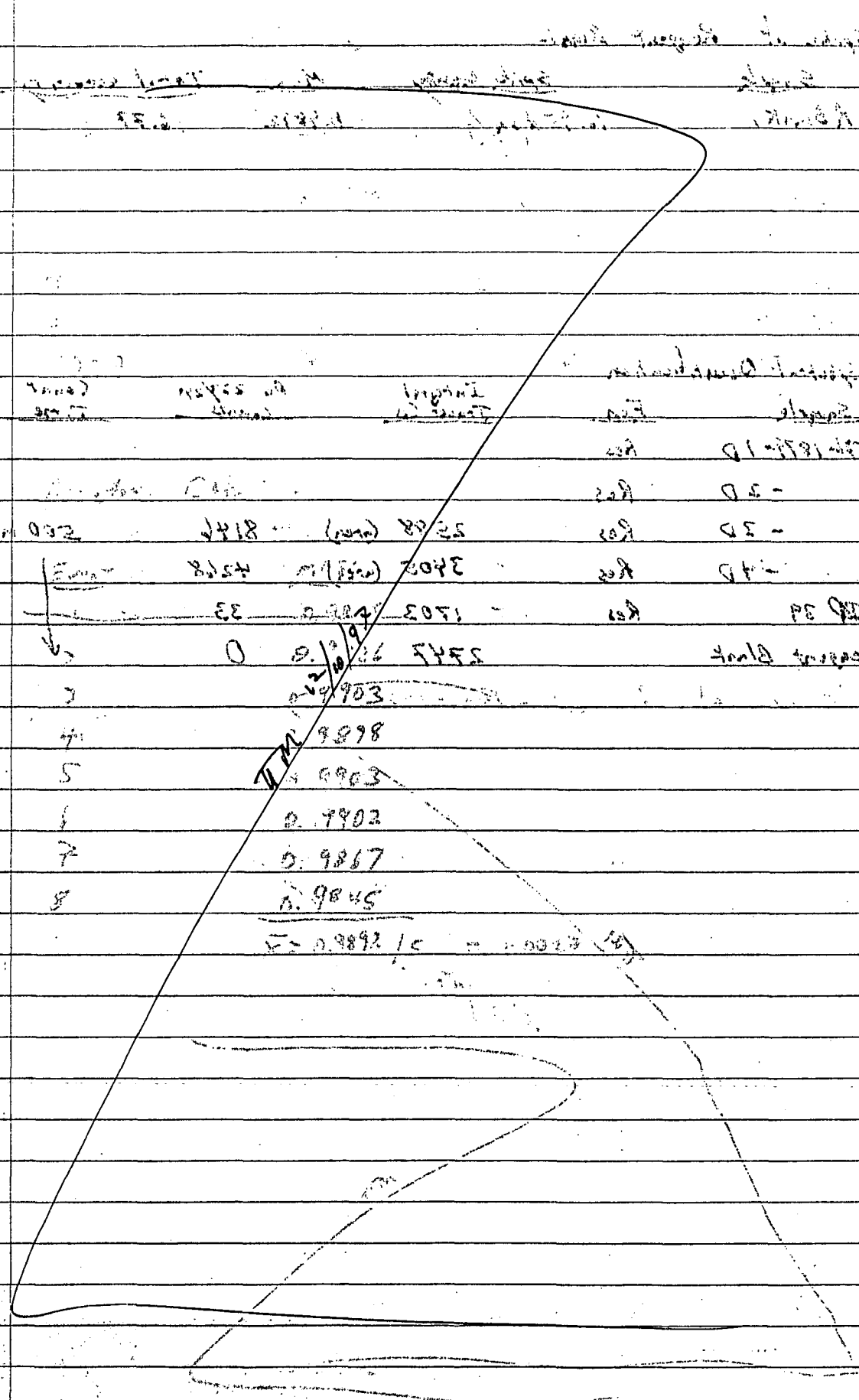
TTM

12/5 Spike of Reagent Blank

Sample	Spike Activity	Mass	Total activity
R Blank 1	16.95 dpm/g	0.9892	16.77

12/10/96 Spectral Quantification

Sample	Fxn	Integral Tracer Cts	Pu-239/241 Counts	Count Time	Tra Di
97L 1879-1D	Res				
-2D	Res				
-3D	Res	2588 (area)	8146	500 m	
-4D	Res	3405 (area)	4268		
SRM 39	Res	1703	33		
Reagent Blank		2747	0		



12/10/17 Mass of Soils

Tare Weights		mass
B	1 L Nalgene	102.0 g
A	500 mL Nalgene	55.0 g
C	250 mL widemouth glass	170.0 g

Sample	Container	Fxn	mass to TA+M	Mass (g)	C/A+B
97L1879-01	1 L Nalgene	B		937.0	
"	500 mL	A		523.0	
"	250 mL	C	17.2	301.5	TM 0.107
02	1L	B		754.5	
"	500	A		411.0	
"	250	C	14.3	357.0	TM 0.185
03	1L	B		1088.0	
	500	A		208.5	
	250	C	16.4	375.0	TM 0.180
04	1L	B		776.0	
04	bag only	A		602.5	
"	250	C	15.8	331.5	TM 0.132
05	1L	B		1068.5	
"	500	A		328.0	
	250	C	18.1	361.0	TM 0.154
06	1L	B		729.5	
"	500	A		316.0	
"	250	C	20.5	286.5	TM 0.124

A = Debris

B = Debris removed, not dried or sieved

C = Dried & Sieved thru 2.36 mm (portion used for analysis)

Sample	Container	Fan	Mass # Tag	Mass (g)	[illegible]
[illegible]	1 L Hopper	B		925.0	
"	500 mL	A		523.0	
"	250 mL	C	17.2	361.5	~~C~~ 0.114
02	1L	B		724.5	
"	500	A		411.0	
"	250	C	14.3	257.0	~~C~~ 0.200
03	1L	B		1085.0	
"	500	A		208.5	
"	250	C	16.4	375.0	~~C~~ 0.194
04	1L	B		776.0	
04	bag only	A		602.5	
"	250	C	15.8	331.5	~~C~~ 0.145
05	1L	B		1068.5	
"	500	A		328.0	
"	250	C	18.1	361.0	~~C~~ 0.169
06	1L	B		729.5	
"	500	A		361.0	
"	250	C	20.5	286.5	~~C~~ 0.146

TM 1/16/98

A = Debris

B = Debris removed not dried or sieved

C = Dried & Sieved thru 2.36 mm (portion used for analysis)

4/16/98 TM Extraction of 97L1879-02
Analysis of Extractants for Am-241 + Pu-238

Aliquot sample	MASS
SOIL BLANK 98-01	3.0372 g
97L1879-002 A	3.0158 g
97L1879-002 B	3.0163 g
97L1879-002 C	3.0187 g
97L1879-002 D	3.0127 g
97L1879-002 E	3.0353 g

4/1/22/98 ① 8 ml of .1 M KNO_3 added to each sample. Samples agitated continuously for 1 hr. at room temperature to remove exchangeable cations. SAMPLES CENTRIFUGED AT 2000 RPM FOR 6 MINUTES. EXTRACTANT FILTERED THROUGH 0.45 MICRON FILTER. RESIDUES RINSED BACK INTO SAMPLE VIALS FROM FILTER PAPER W/ APPROXIMATELY 5 ml D.I. WATER. SAMPLE VIALS VORTEXED FOR APPROX. 15 SECS., THEN CENTRIFUGED AT 2000 RPM FOR 6 MINUTES. RESULTING RINSE WATER FILTERED INTO EXTRACTANT. RESIDUES AGAIN RINSED FROM FILTER PAPER INTO SAMPLE VIALS W/ APPROXIMATELY 1.5 ml D.I. WATER. EXTRACTANTS ACIDIFIED W/ .1 ml CONC. HNO_3 EACH. LABELED AS "EXCHANGEABLE".

4/29/98 ② 8 ml OF 1 M NaOAc ADDED TO RESIDUES OF STAGE 1 AT ROOM TEMPERATURE. ALL SAMPLES VERIFIED TO BE AT pH 5.00. EXTRACTION CARRIED OUT FOR 5 HRS. UNDER CONTINUOUS AGITATION TO REMOVE CARBONATES. SAMPLES CENTRIFUGED AT 2000 RPM FOR 6 MINUTES TO STOP EXTRACTION. EXTRACTANT FILTERED THROUGH 0.45 MICRON FILTER. RESIDUES RINSED BACK INTO SAMPLE VIALS FROM FILTER PAPER

W/ APPROXIMATELY 5 ml D.I. WATER. SAMPLE VIALS VORTEXED FOR APPROX. 5 SECS., THEN CENTRIFUGED AT 2000 RPM FOR 6 MINS. RESULTING RINSE WATER FILTERED INTO EXTRACTANT. RESIDUES AGAIN RINSED FROM FILTER PAPER BACK INTO SAMPLE VIALS W/ APPROX. 5 ml DI WATER. EXTRACTANTS ACIDIFIED W/ 1 ml CONC. HNO_3 EACH. LABELLED AS "CARBONATES".

2/5/98 (3) 20 ml OF 0.04 M $NH_2OH \cdot HCl$ IN 25% v/v HOAc ADDED TO RESIDUES OF STAGE 2. SAMPLE VIALS PLACED IN 96 ± 3 °C WATER BATH WITH OCCASIONAL AGITATION FOR 6 HRS. SAMPLES CENTRIFUGED AT 2000 RPM FOR 6 MINUTES TO STOP EXTRACTION. EXTRACTANT FILTERED THROUGH 0.40 MICRON MEMBRANE FILTER. RESIDUES RINSED FROM FILTER BACK INTO SAMPLE VIALS W/ APPROXIMATELY 5 ml DI WATER. SAMPLE VIALS VORTEXED FOR APPROX. 5 SECS., THEN CENTRIFUGED AT 2000 RPM FOR 6 MINS. RESULTING RINSE WATER FILTERED INTO EXTRACTANT. RESIDUES AGAIN RINSED FROM FILTER BACK INTO SAMPLE VIALS W/ APPROX. 5 ml DI WATER. EXTRACTANTS ACIDIFIED W/ 1 ml CONC. HNO_3 EACH. LABELLED AS "OXIDES / HYDROXIDES."

2/12/98 (4i) 3 ml OF 0.02 M HNO_3 AND 5 ml OF 30% H_2O_2 AT pH 1.83 ADDED TO EACH OF RESIDUES FROM STAGE 3. SAMPLE VIALS PLACED IN 85 ± 2 °C WATER BATH WITH OCCASIONAL AGITATION FOR 2 HRS. SAMPLES CENTRIFUGED AT 2000 RPM FOR 6 MINUTES TO STOP EXTRACTION. EXTRACTANT FILTERED THROUGH 0.40 MICRON MEMBRANE FILTER. ~~EXCESSIVE AMOUNTS OF DI WATER WERE REQUIRED TO WASH RESIDUE FROM FILTER FOR SAMPLES A+B, SO THESE SAMPLES WERE AGAIN CENTRIFUGED AND FILTERS FOR A+B AND REMAINING SAMPLES FILTERS WERE RETURNED TO SAMPLE VIALS.~~ RESIDUES RINSED FROM FILTER BACK INTO SAMPLE VIALS W/ APPROXIMATELY 5 ml DI WATER. SAMPLE VIALS

2/13/98

VORTEXED FOR APPROXIMATELY 5 SECS, THEN CENTRIFUGED AT 2000 RPM FOR 6 MINS. RESULTING RINSE WATER FILTERED INTO EXTRACTANT. RESIDUES FROM SOIL BLANK 98-01 RINSED FROM FILTER BACK INTO SAMPLE VIAL. EXCESSIVE AMOUNTS OF DI WATER WERE REQUIRED TO RINSE RESIDUES FROM FILTERS FOR SAMPLES: 97L1879-002A AND -002B, SO THESE SAMPLES WERE CENTRIFUGED AND REFILTERED. FILTERS FROM ALL 97L1879 SAMPLES WERE THEN RETURNED TO SAMPLE VIALS.

EXTRACTANTS ACIDIFIED W/ 1 ml CONC. HNO_3 EACH. LABELED AS "ORGANICS (i)"

02/27/98 STANDARDS DILUTION

BALANCE: METTLER AK160 SERIAL # A70276

CALIBRATED 4-9-2-98 (STANDARD)

CHECKED WITH DENVER INST. CO. MASS SET, SER. # 97-J35874

	ACTUAL MASS	BALANCE READOUT
	10 mg	0.0100 g
	100 mg	0.1000 g
	1 g	1.0000 g
	10 g	9.9998 g

Pu-242 DILUTION: INITIAL CONCENTRATION = 25.26 Bq/g

125 ml MASS OF BOTTLE (W/ LID): 116.2454 g

+ ~1 g Pu-242: 117.3358 g MASS Pu242: 1.0904

+ 100 ml 2.8 M HNO_3: 125.3884 g MASS SOLN: 109.143

30 ml MASS OF BOTTLE (W/ LID): 10.9552 g

+ ~4 ml Pu-242: 15.2999 g MASS Pu242: 4.3447 g

+ 20 ml 2.8 M HNO_3: 26.7568 g MASS SOLN: 25.8016

Am-243 DILUTION: INITIAL CONC. = 36.27 Bq/g TRACK # RH26

125 ml BOTTLE MASS: 16.2878 g

JDW 27 FEB 97

+ ~0.5 ml Am-243 STD: 16.7949 g MASS STD: 0.5071 g

+ ~100 ml, 1 M HNO_3: 118.7194 g MASS SOLN: 102.4316 g

FINAL Am-243 CONC: $\left(\frac{36.27\ Bq}{g}\right)\left(\frac{0.5071\ g}{102.4316\ g}\right) = 0.1796\ Bq/g$

DESIGNATED TRACK # RH261A

30 ml BOTTLE MASS: 10.9328 g

+ ~4.5 ml Am-243 STD: 15.5370 g MASS STD: 4.6042 g

+ ~20 ml 1 M HNO_3: 35.1810 g MASS SOLN: 24.2482 g

FINAL Am-243 CONC: $\left(\frac{36.27\ Bq}{g}\right)\left(\frac{4.6042\ g}{24.2482\ g}\right) = 6.8869\ Bq/g$

DESIGNATED TRACK # RH261B

Pu-242 DILUTION (FROM DATA PREVIOUS PAGE)

INITIAL CONC: 28.26 Bq/g TRACK # RH260

125 ml BOTTLE DILUTION

FINAL Pu-242 CONC: $\left(\frac{28.26\ Bq}{g}\right)\left(\frac{1.0904\ g}{109.1430\ g}\right) = 0.2823\ Bq/g$

DESIGNATED TRACK # RH260A

30 ml BOTTLE DILUTION

FINAL Pu-242 CONC: $\left(\frac{28.26\ Bq}{g}\right)\left(\frac{4.3447\ g}{25.8016\ g}\right) = 4.7587\ Bq/g$

DESIGNATED TRACK # RH260B

3/30/98 TM Mass accounting of Sample 97L1879-002
Sieved through 2.00 mm mesh, placed in 1L Nalgene bottle and shaken for several minutes
Riffle split through Humboldt Model H-3973 serial # 2/78 three passes to obtain portion for analysis.

562 grams unused portion
80 grams for analysis
642 grams total

4/1/98 TM Aliquoting of samples for Reproducibility Study

10g series	dish	dish mass	gross mass	soil mass
10-1	1	43.5161	53.5800	
10-2	2	42.4297	52.5177	
10-3	3	45.5611	55.4378	
10-4	4	44.7913	54.5898	
10-5	5	44.5868	54.8215	

3g series	dish	dish mass	gross mass	soil Mass
3-1	6	44.0400	47.0790	3.0390g
3-2	7	42.2344	45.3012	3.0668
3-3	8	44.2046	47.5228	3.3182
3-4	9	47.6451	50.9542	3.3091
3-5	10	43.7094	46.7317	3.0223

1g series	dish	mass	gross mass	Net
1-1	11	44.8678	45.9006	1.0328
1-2	12	43.2525	44.2789	1.0264
1-3	13	43.1086	44.1157	1.0071
1-4	14	44.4721	45.4933	1.0212
1-5	15	43.5195	44.5350	1.0155

4/1/98 Balance Check Mettler HK 160 SNR A70276

Weight	Balance Reading
1g	1.0000
0.1 g	0.1000
10 g	9.9997
~~100g~~	

QC Samples

Matrix Spike
~~LCS~~ - 3g of 97L1879-02 spiked with Pu-239 and Am-241
Blank is 3g of silica sand
LCS is 3g of silica sand spiked with Pu-239 and Am-241

Aliquots

	Matrix Spike (disk 16)	Blank (disk 17)	LCS (disk 18)
~~tare~~	41.1704	4/1/98 ~~42.8434~~ 3	43.3115
gross	44.2371	46.8711	46.3613
net			

Sample Spiking

Sample	disk	Pu-242 tracer mass 4.7587 Bq/g RH-260B dil. 1	Am-243 tracer mass RH-261A ~~0.176~~ 4/1/98 0.1796 Bq/g
10-1	1	1.0895 g	1.0166
10-2	2	1.0861 g	1.0212
10-3	3	1.0864 g	1.0177
10-4	4	1.0820 g	1.0224
10-5	5	1.0825 g	1.0140

4.4

[illegible] Balance Check Mettler HK 160 SNR A[illegible]

Weight	Balance Reading
1g	1.0000
0.1g	0.1000
10g	9.9997

QC Samples

Matrix Spike

~~LCS~~ - 3g of 9711839-02 spiked with Pu-239 and Am-241

Blank is 3g of silica sand

LCS is 3g of silica sand spiked with Pu-239 and Am-241

Aliquots

	Matrix Spike (dish 16)	Blank (dish 17)	LCS (dish 18)
tare	41.1704	41.8434	43.3115
grass	44.2371	46.8711	46.3613
net			

Sample Spiking

Sample	dish	Pu-242 Tracer mass 4.7587 Bq/g	RH-2608 dil. 1	Am-243 tracer mass 0.1796 Bq/g	RH-261A dil. 1
10-1	1	1.0895 g		1.0116	
10-2	2	1.0861 g		1.0212	
10-3	3	1.0864 g		1.0177	
10-4	4	1.0820 g		1.0224	
10-5	5	1.0825 g		1.0140	

4/1/98 Spiking of Samples (cont.)

Sample	dish #	Pu-242 tracer mass	SRM 4334F 16.95 dpm/g	Am-243 tracer mass	RH-2 0.1796 Bq/
3-1	6	5.2547		1.0094	
3-2	7	5.2412		1.0078	
3-3	8	5.2399		1.0111	
3-4	9	5.2134		1.0071	
3-5	10	5.3478	RH 260A 0.2823 Bq/g	1.0144	

4/2/98 ALL 971479-002 EXTRACTIONS (THROUGH ORGANICS (i)) AND BLANK 98-01 EXTRACTIONS TRANSFERRED TO SAMPLE TUBES AND BROUGHT TO 30 ml TOTAL VOLUME WITH 1 M HNO_3.

Addition of Spike to Remaining Samples

Sample	dish #	Pu-242 tracer mass	RH 260A 0.2823 Bq/g	Am-243 tracer mass	RH-261 0.1796 Bq/g
1-1	11	1.0900		1.0263	
1-2	12	1.0878		1.0207	
1-3	13	1.0849		1.0200	
1-4	14	1.0861		1.0206	
1-5	15	1.0838		1.0195	
Matrix Spike (MS1)	16	1.0811		1.0211	
Blank	17	1.0754		1.0220	
LCS (LCS1)	18	~~1.08~~ TM 4/6/98 1.0770		1.0213	

4/3/98 3 ml OF ALL 971479-002 AND SOIL BLANK 98-01 EXTRACTION TRANSFERRED TO 15 ml SAMPLE TUBES AND DILUTED TO 10 ml TOTAL VOLUME WITH 1 M HNO_3, IN PREPARATION FOR ICP METALS ANALYSIS. REMAINDER OF EXTRACTIONS RETURN TO BEAKERS FOR [illegible]

4/9/98 SPIKING:

BALANCE CHECK ON METTLER HK160 SER # A70276
W/ STANDARDS TRACEABLE TO NIS SER # 97-J35874-8

STANDARD MASS	MEASURED MASS
10 g	9.9999 g
1 g	1.0000 g
100 mg	0.1000 g
10 mg	0.0101 g

BASED ON EARLIER WORK, ESTIMATES OF TRACER ACTIVITY REQUIRED FOREACH FRACTION ARE AS FOLLOWS:

FRACTION	SAMPLE ACTIVITY (Bq/g)	ACTIVITY REQ'D FOR 3g SAMPLE (Bq)
EXCHANGEABLE	9.52×10^{-3}	2.56×10^{-2}
CARBONATE	1.96×10^{-2}	5.89×10^{-2}
SESQUIOXIDE	3.67×10^{-1}	1.10
ORGANIC	3.59	10.78
RESIDUAL	9.78	29.33

Am-243: STD RH 261A 0.1796 Bq/g in 1.0 M HNO_3

EXTRACTANT	VOL STD ADDED	TINIT. STD MASS(g)	FINAL STD MASS(g)	MASS STD ADDED(g)	ACTIVITY ADDED (Bq)
EXCHANGEABLE:					
98-01 SOIL BLANK	0.5 ml	100.3836	99.8738	0.5099	9.16×10^{-2}
971879-002A	0.5 ml	99.8738	99.3642	0.5096	9.15×10^{-2}
971879-002B	0.5 ml	99.3642	98.8531	0.5111	9.18×10^{-2}
971879-002C	0.5 ml	98.8531	98.3419	0.5112	9.18×10^{-2}
971879-002D	0.5 ml	98.3419	97.8312	0.5107	9.17×10^{-2}
971879-002E	0.5 ml	97.8312	97.3195	0.5117	9.19×10^{-2}
~~971879-002~~ QDUP					

4/9/93 SPIKING:

BALANCE CHECK ON METTLER HN-100 PER # A70276
W/ STANDARDS TRACEABLE TO NIS PER # 97-135774-E

STANDARD MASS	MEASURED MASS
10 g	9.9999 g
1 g	1.0000 g
100 mg	0.1000 g
10 mg	0.0101 g

BASED ON EARLIER WORK, ESTIMATES OF TRACER ACTIVITY REQUIRED FOR EACH FRACTION ARE AS FOLLOWS:

FRACTION	SAMPLE ACTIVITY (Bq/g)	ACTIVITY REQ'D FOR 3g SAMPLE (Bq)
EXCHANGEABLE	5.52×10^{-3}	2.56×10^{-2}
CARBONATE	1.96×10^{-2}	5.89×10^{-2}
SESQUIOXIDE	3.67×10^{-1}	1.10
ORGANIC	3.59	10.78
RESIDUAL	9.78	29.33

Am-243: STD RH 261A 0.1796 Bq/g in 1.0 M HNO_3

EXTRACTANT	VOL STD ADDED	INIT. STD MASS(g)	FINAL STD MASS(g)	MASS STD ADDED(g)	ACTIVITY ADDED(Bq)
EXCHANGEABLE:					
1-01 SOLL BLANK	0.5 ml	100.5836	99.8738	0.5098	9.16×10^{-2}
1879-002A	0.5 ml	99.8738	99.3642	0.5096	9.15×10^{-2}
1879-002B	0.5 ml	99.3642	98.8531	0.5111	9.18×10^{-2}
1879-002C	0.5 ml	98.8531	98.3419	0.5112	9.18×10^{-2}
1879-002D	0.5 ml	98.3419	97.8312	0.5107	9.17×10^{-2}
1879-002E	0.5 ml	97.8312	97.3195	0.5117	9.19×10^{-2}

4/9/98 Am-243 SPIKING (CONT'D)

EXTRACTANT	VOL STD ADDED	INIT. STD MASS(g)	FINAL STD MASS(g)	MASS STD ADDED(g)	ACTIVITY ADD
CARBONATES:					
SOIL BLANK 98-01	0.5 ml	97.3195	96.8070	0.5125	9.20×10^{-1}
97L1879-002A	0.5 ml	96.8070	96.2938	0.5132	9.22×10^{-1}
97L1879-002B	0.5 ml	96.2938	95.7808	0.5130	9.21×10^{-1}
97L1879-002C	0.5 ml	95.7808	95.2682	0.5126	9.21×10^{-1}
97L1879-002D	0.5 ml	95.2682	94.7559	0.5123	9.20×10^{-1}
97L1879-002E	0.5 ml	94.7559	94.2436	0.5123	9.20×10^{-1}
SESQUIOXIDE:					
SOIL BLANK 98-01	6 ml	94.2436	88.1088	6.1348	1.10
97L1879-002A	6 ml	88.1088	81.9737	6.1351	1.10
97L1879-002B	6 ml	81.9737	75.8340	6.1397	1.10
97L1879-002C	6 ml	75.8340	69.7019	6.1321	1.10
97L1879-002D	6 ml	69.7019	63.5763	6.1256	1.10
97L1879-002E	6 ml	63.5763	57.4564	6.1199	1.10

Pu-242: STD RH 260A 0.2823 Bq/g in 2.8 M HNO_3

EXTRACTANT	VOL STD ADDED	INIT. STD MASS(g)	FINAL STD MASS(g)	MASS STD ADDED(g)	ACTIVITY ADDE
EXCHANGEABLE:		~~111.3553~~ IPW			
SOIL BLANK 98-01	0.5 ml	111.3553	110.8134	0.5239	1.48×10^{-1}
97L1879-002A	0.5 ml	110.8134	110.2733	0.5591	1.58×10^{-1}
97L1879-002B	0.5 ml	110.2733	109.7338	0.5395	1.52×10^{-1}
97L1879-002C	0.5 ml	109.7338	109.1937	0.5401	1.52×10^{-1}
97L1879-002D	0.5 ml	109.1937	108.6535	0.5402	1.52×10^{-1}
97L1879-002E	0.5 ml	108.6535	108.1125	0.5410	1.53×10^{-1}

4/9/98 Pu-242 SPIKING (CONT'D)

EXTRACTANT	VOL STD ADDED	INIT. STD MASS (g)	FINAL STD MASS (g)	MASS STD ADDED (g)	ACTIVITY ADDE
CARBONATE:					
SOIL BLANK 98-01	0.5 ml	108, 1125	107. 5732	0, 5393	1.52×10^{-1}
97L1879-002A	0.5 ml	107. 5732	107. 0333	0.5399	1.52×10^{-1}
97L1879-002B	0.5 ml	107. 0333	106. 4921	0.5412	1.53×10^{-1}
97L1879-002C	0.5 ml	106, 4921	105. 9523	0.5398	1.52×10^{-1}
97L1879-002D	0.5 ml	105. 9523	105. 4120	0.5403	1.53×10^{-1}
97L1879-002E	0.5 ml	105, 4120	104. 8732	0, 5388	1.52×10^{-1}
SESQUIOXIDE:					
SOIL BLANK 98-01	4 ml	104. 8732	100. 5370	4, 3362	1, 22
97L1879-002A	4 ml	100, 5370	96. 1981	4, 3389	1, 22
97L1879-002B	4 ml	96. 1981	91. 8611	4, 3370	1, 22
97L1879-002C	4 ml	91. 8611	87. 5267	4, 3344	1, 22
97L1879-002D	4 ml	87. 5267	83. 1921	4, 3346	1, 22
97L1879-002E	4 ml	83. 1921	78. 8609	4, 3312	1, 22

TM

4/9/98

4/9/98 Transfer & Dilution of Standards

TM Pu-239 37.9 TM
SRM 4330A ~~37.24~~ Bq/g
CSM # RH-264

Am-241
SRM 4322B 39.24 Bq/g
CSM # RH-265

Transfer to Dilution Vial →

gross	10.7612 g	RH-264B
tare	9.6880 g	
net	1.0732	

	11.1789 g	RH265B
	10.1533 g	
	1.0256	

Transfer to Vial of Original →

gross	10.68 g	
tare	5.22 g	
net	5.46 g	

	10.35 g	
	5.22 g	
	5.13 g	

Dilution of RH 264B

gross	38.0115 g	Diluted w
tare	9.6880 g	2 M HNO_3
net	28.3235 g	

Dilution of RH 265B

	36.5659 g	Diluted w
	10.1533 g	1 M HNO_3
	26.4126 g	

Activity of Dilution

RH-264B

$$\frac{1.0732\ g}{28.3235\ g} * 37.9\ Bq/g$$

$$= \boxed{1.436\ Bq/g}$$

RH-265B

$$\frac{1.0256\ g}{26.4126\ g} * 39.24\ Bq/g$$

$$= \boxed{1.5237\ Bq/g}$$

4/9/98

TM

4/13/98 Spiking of QC samples

Sample	Pu-239 RH-264B	1.436 Bq/g	Am-241 RH-265B	1.524 Bq/g
~~gross~~ LCS1 (Blank Spike)	~~28.1444~~	1.0511 g	1.0113	
~~true~~ TM 4/13/98 ~~Matrix Spike MS~~	5.2675	g	1.0114	
~~not~~ Matrix Spike (MS1)				

Balance Check Mettler 160

mass	readout
100 mg	0.1000
1 g	1.0001
10 g	10.0000

4/16/98 (4ii) 3ml of 30% H_2O_2 (ADJUSTED TO pH 1.98 W/ 0.1 M HNO_3 ADDED TO EACH OF RESIDUES FROM STAGE 4i. SAMPLE VIALS PLACED IN 85 ± 2 °C WATER BATHS WITH OCCASIONAL AGITATION FOR 3 HRS.

SAMPLES CENTRIFUGED AT 4000 RPM FOR 6 MINS TO STOP EXTRACTI EXTRACTANT FILTERED THROUGH 0.40 MICRON MEMBRANE FILTERS. MEMBRANES RETURNED TO SAMPLE VIALS.

4/17/98 RESIDUES FROM EXTRACTION 4ii RINSED W/ APPROXIMATELY 5 ML DI WATER EACH. SAMPLE VIALS CENTRIFUGED AT 4000 RPM FOR 6 MINUTES. RESULTING RINSE WATER ~~RINSED~~ DPW FILTERED INTO EXTRACTANT (RINSE & EXTRACTION 4ii ABOVE WERE BOTH COMBINED W/ EXTRACTION 4i).

(4iii) RESIDUES FROM EXTRACTION 4ii COMBINED W/ 5 ml OF 3.2 M NH_4OAc IN 20% (V/V) HNO_3, DILUTED TO 20 ml W/ DI WATER SAMPLES AGITATED CONTINUOUSLY FOR 20 MINS. AT ROOM TEMP. SAMPLES CENTRIFUGED AT 4000 RPM FOR 6 MINS TO STOP EXTRACTION. EXTRACTANT FILTERED THROUGH 0.40 MICRON MEMBRANE FILTER AND COMBINED W/ EXTRACTANTS FROM STEPS 4i AND 4ii. RESIDUES RINSED W/ APPROX. 5 ML DI WATER, VORTEXED FOR 5 SECS., CENTRIFUG AT 2000 RPM FOR 6 MINS, RINSE WATER FILTERED AND COMBINED W/ EXTRACTANT 1 ml CONC. HNO_3 ADDED TO ACIDIFY EXTRACTANTS.

4/22/98 Aliquots of 97L1879-002 for Redox work

	R-1	R-2	R-3	R-4	R-5
gross					
tare	16.8446				
net					

4/23/98 ORGANIC EXTRACTANTS FROM STEP 4 MISTAKENLY "COOKED DOWN" PRIOR TO SPIKING. ALL ORGANIC EXTRACTANTS REDISOLVED IN 15 ml CONC. HNO_3. 3ml ALIQUOTS OF ORGANIC EXTRACTANTS REMOVED TO 15 ml SAMPLE TUBES AND DILUTED TO 10 ml TOTAL VOLUME WITH 1M HNO_3, IN PREPARATION FOR ICP METALS ANALYSIS.

(5) RESIDUES FROM EXTRACTIONS 1-4 WASHED FROM SAMPLE VIALS INTO TEFLON BEAKERS W/ APPROXIMATELY 10 ml OF D.I. WATER EACH. 25 ml HF AND 5 ml $HClO_4$ ADDED TO EACH BEAKER (5:1 RATIO).

4/24/98 SPIKING:

BALANCE CHECK ON A&D ER-60A SER# 3403338 W/ STDS TRACEABLE TO NIS SER # 97-J3587A-8

STD MASS	MEASURED MASS
M.P. 10 g	10.0000 g
1 g	1.0000 g
100 mg	0.1000 g
10 mg	0.0096 g

BASED ON EARLIER PIXWORK, ESTIMATES OF TRACER ACTIVITY REQUIRED FOR REMAINING FRACTIONS ARE AS FOLLOWS:

FRACTION	SAMPLE ACTIVITY (Bq/g)	ACTIVITY REQ'D FOR 3g SAMPLE
ORGANIC	3.59	10.78
RESIDUAL	9.78	29.33

STD RH261A 0.1796 Bq/g IN 1.0 M HNO_3

4/24/98 Am-243 : ~~STD RH261B 6.8869 Bq/g IN 1.0 M HNO_3~~ SPW 4/24/98

EXTRACTANT:	VOL STD ADDED	INIT. STD MASS (g)	FINAL STD MASS (g)	MASS STD ADDED (g)	ACTIVITY ADDED (
ORGANIC :					
98-01 SOIL BLANK	1 ml	57.4532	56.4263	1.0269	0.1844
97L1879-002A	1 ml	56.4263	55.4033	1.0230	0.1837
97L1879-002B	1 ml	55.4033	54.3784	1.0249	0.1841
97L1879-002C	1 ml	54.3784	53.3533	1.0251	0.1841
97L1879-002D	1 ml	53.3533	52.3283	1.0250	0.1841
97L1879-002E	1 ml	52.3283	51.3043	1.0240	0.1839
RESIDUAL :					
98-01 SOIL BLANK	1 ml	51.0304	50.2814		
97L1879-002A	1 ml	50.2814	49.2227		
97L1879-002B	1 ml	49.2227	48.1836		
97L1879-002C	1 ml	48.1836	47.1452		
97L1879-002D	1 ml	47.1452	46.1083		
97L1879-002E	1 ml	46.1083	45.0705		

P_u-242 : STD RH260B 4.7587 Bq/g IN ~~1.0 M~~ SPW 2.8 M HNO_3

EXTRACTANT	VOL STD ADDED	INIT. STD MASS (g)	FINAL STD MASS (g)	MASS STD ADDED (g)	ACTIVITY ADDED (
ORGANIC :					
98-01 SOIL BLANK	1 ml	32.4015	31.3029	1.0986	5.228
97L1879-002A	1 ml	31.3029	30.2098	1.0931	5.202
97L1879-002B	1 ml	30.2098	29.1164	1.0934	5.203
97L1879-002C	1 ml	29.1164	28.0223	1.0941	5.207
97L1879-002D	1 ml	28.0223	26.9302	1.0921	5.197
97L1879-002E	1 ml	26.9302	25.8385	1.0917	5.195

4/24/98 Pu-242 SPIKING (CONT'D)

EXTRACTANT	VOL STD ADDED	INIT. STD MASS(g)	FINAL STD MASS(g)	MASS STD ADDED(g)	ACTIVITY ADDE
RESIDUAL:					
98-01 SOIL BLANK	1 ml	25.8385	24.7478	1.0907	5.190
97L1879-002A	1 ml	24.7478	23.6588	~~1.0937~~ 1.0890 TM 4/15/98 ~~5.202~~	5.
97L1879-002B	1 ml	23.6588	22.5714	1.0874	5.175
97L1879-002C	1 ml	22.5714	21.4884	1.0830	5.154
97L1879-002D	1 ml	21.4884	20.4044	1.0840	5.158
97L1879-002E	1 ml	20.4044	19.3227	1.0817	5.148

5/20/98 97L1879-002 AND 98-01 SOIL BLANK EXCHANGEABLE FRACTIONS REDISOLVED IN 5ml CONCENTRATED HNO_3. SAMPLES RINSED FROM BEAKERS INTO SAMPLE VIALS WITH DI WATER. SAMPLES THEN DILUTED TO 30 ml WITH DI WATER. SODIUM NITRITE ADDED IN EXCESS TO EACH VIAL. 0.5 ml 20 mg/L Fe^{3+} AS CHLORIDE IN 0.5 N HCl ADDED TO EACH SAMPLE VIAL. EACH SAMPLE MADE BASIC BY ADDITION OF NH_4OH (CONC.) TO 45 ml. SAMPLE TUBES CENTRIFUGED AT 4000 RPM FOR 6 MINS. SUPERNATE POURED OFF AS WASTE. SAMPLES REDISOLVED IN 5ml 12 M HCl AND 15 ml 9 M HCl

97L1879-002 AND 98-01 SOIL BLANK OXIDES/HYDROXIDES FRACTIONS REDISOLVED IN 10 ml CONCENTRATED HNO_3. SAMPLES RINSED FROM BEAKERS INTO SAMPLE VIALS WITH DI WATER. SAMPLES THEN DILUTED TO 30 ml WITH DI WATER. SODIUM NITRITE ADDED IN EXCESS TO EACH SAMPLE VIAL. 0.5 ml 20 mg/L Fe^{3+} AS CHLORIDE IN 0.5 N HCl ADDED TO EACH SAMPLE VIAL. EACH SAMPLE MADE BASIC BY ADDITION OF NH_4OH (CONC TO BRING TOTAL VOLUME TO 45 ml. SAMPLE TUBES CENTRIFUGED AT 4000 RPM FOR 6 MINS. SUPERNATE POURED OFF AS WASTE. SAMPLES REDISOLVED IN 5 ml 12 M HCl AND 15 ml 9 M HCl

5/21/98 9711879-002 AND 98-01 SOIL BLANK CARBONATE FRACTIONS REDISOLVED IN 10 ml CONCENTRATED HNO_3. SAMPLES RINSED FROM BEAKERS INTO SAMPLE VIALS WITH DI WATER, THEN DILUTED TO 30 ml TOTAL VOLUME WITH DI WATER. SODIUM NITRITE ADDED IN EXCESS TO EACH SAMPLE VIAL. 0.5 ml 20 mg/L Fe^{3+} AS CHLORIDE IN 0.5 N HCl ADDED TO EACH SAMPLE VIAL. EACH SAMPLE MADE BASIC BY ADDITION OF CONCENTRATED NH_4OH TO BRING TOTAL VOLUME TO ~45ml. SAMPLE TUBES CENTRIFUGED AT 4000 RPM FOR 6 MINS. SUPERNATE POURED OFF AS WASTE. SAMPLES REDISOLVED IN 15 ml 12 M HCl AND 15 ml 9 M HCl.

6/15/98 Integration of Spectral Peaks

TM

Sample	~~Filename~~ fxn	Element ~~fxn~~	~~Element~~ Tracer Counts	Analyte Counts	Count Time	T[illegible] B[illegible]
Soil Blank 98-01	Exchang.	Pu	2501	19	1000m	0.14
	Carbonate		2824	7		0.15
	Sesquiox.		22348	17		TM 6/15/98 ~~0~~ 1.[illegible]
	Organic		TM 6/15/98 ~~92553~~ 93682	130		5.2
	Residual		34956	72		5.19
97L1879-02 A	Exchange	Pu	1170	30	1000m	0.15[illegible]
B			1726	41		0.152
C			2616	57		0.152
D			2492	48		0.152
E			2520	35		0.153
97L1879-02 A	Carbonate	Pu	2727	832	1000m	0.152
B			2824	735		0.153
C			2792	749		0.152
D			2860	773		0.153
E			2716	664		0.152
					1000m	
97L1879-02 A	Oxides	Pu	22218	11429		1.22
B			22477	14031		1.22
C			23861	11010		1.22
D			22510	13520		1.22
E			23089	11880		1.22
97L1879-02 A	Organics	Pu	94959	191885	1000m	5.202
B			94244	221792		5.203
C			96433	175321		5.207
D			104733	210402		5.197
E			99173	242566		5.195

6/15/98 Spectral Integration (cont)

ITM	Sample		Fxn	Element	Tracer Counts	Analyte counts	Count time	Tracer Rg
	97L1879-02	A	Residual	Pu	71636	90330	1000m	5.182
		B			59636	71443		5.175
		C			70749	77808		5.154
		D			21526	34315		5.158
		E			78446	91919		5.148
	NR01		NA	Pu	73271	1231	1000m	5.154
	NR02				13217	337		0.921
	NO02				9599	1133		0.615
	NR02B				14691	841		0.918
	NCS01				15647	1069		0.306
	SCS01				14915	3842		5.465
	Elec. Wash.				2553 (area)	50406		0.307
	BLK-01				5913	70		0.307
	BLK-12				5838	2		0.306
	BLK-13				5313	44		0.306
	NR01		NA	Am	99022	364	1000m	7.05
	SCS01				139987	778		
	NCS01				12990	1111		
	Elec. Wash				1039 (area)	4208		
	BLK-01				3015	31		
	BLK 12				3254	19		
	BLK 13				3123	19		
	97L1879-02	A	Organic	Am	1541 (area)	23108 (area)		~~0.0915~~
		B			1950 (area)	28607 (area)		~~0.0918~~
		C			1161 (area)	32906 (area)		~~0.0918~~
		D			605 (area)	29574 (area)		~~0.0917~~
		E			1404 (area)	29649 (area)		~~0.0919~~
	BLK 98-01				*10	31 ITM 6/15/98		~~0.0916~~

6/24/98 Aliquot of Metals Fraction

Sample		Total Mass	Mass of Metals fxn	fraction of Total
97L1879-02	~~3-1~~ ~~3-5~~	153.5g	16.6g	
	6/24/98 3-2	173.6g	15.8g	
	3-3	170.2g	16.3g	
	3-4	178.1g	16.3g	
	3-5	172.3g	15.8g	

Balance Check Mettler BD 1201

Mass	Reading	OK?
1g	1.0	
10g	10.0	
200g	200.0	

~~0.184~~

~~0.1844~~ 0.1837

0.1841

TDM 6/25/98 0.1841

0.1841

0.1839

0.1844

64

7/7/98 TM

Aliquots taken of Soil 97L1879-02 for Sequential Extraction and Subsequent Pu-239/240 + Am-241 analysec

Sample	Gross wt	Tare	Net
02 - F	16.1671	13.2240	2.943g
02 - G	16.1970	13.2484	2.949g
02 - H	16.2302	13.1825	3.048g
02 - MS2	16.3017	13.27~~22~~07 TM 7/7/98	
Blank 98-02	16.3057	13.2948	3.0109
LCS2	16.1962	13.2542	2.9420

Balance Check Mettler HK 160

Mass	Readout
10 mg	0.0101
100 mg	0.1000
1g	1.0001
10g	10.0000

Spiking of MS2 + LCS

MS2 is 3g of Sample 97L1879-02

LCS is 3g of blank silica at 140 mesh

Sample	RH264B Pu-239 1.436Bq/g	RH-265B Am-241 1.5237Bq/g
MS2	5.3301g	5.1154g
LCS	1.0615g	1.0217g

64

7/7/98 Exchangeable Extraction

Process Started: 4:01 PM Extraction ended: 5:05 PM

Samples filtered through 0.4 μ membrane. No rinse performed since ~~ratio~~ ratio of Am/Pu is information desired.

7/8/98 Carbonate Fraction

Started: 1:02 PM Ended: 6:10 PM

Spiking of Exchangeable Fraction

Sample	Mass Pu-242 (RH260A)	Mass Am243 (RH-261A)
Blank 98-02	1.0986 g	1.0235 g
F	1.0843 g	1.0194 g
G	1.0818 g	1.0065 g
H	1.0748 g	1.0124 g
LCS2	1.0771 g	1.0217 g
MS2	1.0725 g	1.0167 g

Balance Check Mettler HK 160

Mass	Readout
10 mg	0.0100
100 mg	0.1000
1 g	1.0001
10 g	10.0000
100 g	

7/9/98 Oxides/Hydroxides Fraction

Started: 10:40 AM Ended: 4:45 PM

7/13/98 Spiking of Carbonate fraction

Sample	mass of RH 260 Pu-242 (0.2823 Bq/g)	mass of RH 261A Am-243 (0.1796 Bq/g)
Blk 98-02	1.0988	1.0164
G	1.0882	0.9827
H	1.0794	1.0164
F	1.0812	1.0201
MS2	1.0825	1.0035
LCS2	1.0800	1.0193

7/14/98 Balance Check Mettler HK 160

25 Mass	1st Readout ~~7/14/98~~	
10 mg	~~0.0101~~	0.0100
100 mg	0.0999	
1 g	1.0000	
10 g	10.0000	
100 g	99.9986	

7/15/98 Spiking of Sesquioxide fraction

Sample	mass of Am-243 RH 261A (6.8869 Bq/g)
Blk 98-02	0.3114 g
F	0.2070 + 0.5157 = 0.7227 g
G	0.2068 + 0.5166 = 0.7234 g
H	0.2077 g
MS2	0.2074 g
LCS2	0.2087 g

7/17/98 Organic fraction

1st extraction	Start: 7/16 9:00 AM	End: 11:00 AM	
2nd extraction	Start: 7/17 9:30 AM	End: 11:30 AM	
3rd extraction	Start: 7/17 3:20 PM	End: 4:00 PM	

7/20/98 Spike Organic fraction

Sample	Mass of Am-243 RH 261 B 6.8869 Bq/g	Activity
BLK 98-02	0.5174 g +	
F	0.5163 g + ~~0.5157~~	determined tha this was ad
G	0.5228 g + 0.5166	Segq. fraction
H	0.5205 g	than organic
MS2	0.5161 g	
LCS2	0.5174 g	

Balance Check Mettler HK 160

mass	readout
~~1 mg~~	0.0100
10 mg	0.1000 TM 7/20/98
100 mg	1.0000
1 g	9.9999
10 g	
100 g	99.9985

Digestion of Residual fraction / Spiking of Samples

Sample	Teflon Beaker #	Am-243 RH-261B Mass 6.8869 Bq/g		Am-243 Activity
BLK 98-02	3	0.5148		
F	9	0.5115	~~0.5115~~ ~~0.5157~~ g	
G	13	~~0.5166~~	0.5129	TM 7/20/98
H	14	0.5120		
MS2	16	0.5125	~~0.5115~~	
LCS2	18	0.5126		

7/21/98 Aliquot for Am analysis of "Total Dissolution" of Soil

Sample	Teflon Beaker #	sample Mass	Am-243 261B	6.886-9 Bq/g	Am-243 Activity
1-6	1	1.0102 g	1.0327 g		7.1121 Bq
1-7	15	0.9774	1.0285 g		7.0832 Bq
B/K 98-03	7	0.9997	0.5158 g		
LCS 3 (blank Spike)	10	1.0042	0.5157 g		

	Pu-239 RH264B	Pu-242 260B	Am-241 RH26
1-6	—	1.0950 g	—
1-7	—	1.0835 g	—
B/K 98-03	—	0.5477 g	—
LCS 3	1.0656 g	0.5453 g	1.0306 g

1/21/98 APHA Data

Sample	Fxn	Element	Count Time Tracer	Tracer Cts	Analyte Cts
97L1879-02, 1-1	-	Pu	1000 m	4762 (area)	135354
	1-2	-	Pu	3717 (area)	227480
	1-3	-		4533 (area)	143717
	1-4	-		3410 (area)	148389
	1-5	-		4142 (area)	153823
	1-Blk	-	Pu	4275 ~~4234 (area)~~ TM 7/27/98	97
	1- LCS	-	Pu	4295	24839
97L1879-02, 3-1		Pu	216.27 ~~1000 m~~	2742 (area)	82200
	3-2			3828	73579
	3-3			4302	102768
	3-4			4098	112915
	3-5		1000 m	17205	362992
	MS1			809	628461
Blank 98-02	Ex	Pu	1000m	5312	575
97L1879-02 F				5587	785
G				5197	712
H				5510	443
MS2				4576 (area)	161521
LCS2				5329	2418
97L1879-02 F	Ex	Am	1000m	3229	40
G				3435	75
H				2155	18
Blank 98-02				2902	39
LCS2				2264	18850
97L1879-02 F Carb		Am	1000 m	2172 ~~2511~~ (area)	978
G				TM 7/27/98 ~~2606~~ 2164	825
				2296	1091
Blk 98-02				2965	19
MS2				1851 (area)	7133 (area)
LCS2				1758 (area)	3195

7/26 Spectral Data APHA

Sample	Fxn	Element	Ct Time	Tracer Counts	Analyte Counts
BLK 98-02	OX	Am	1000m	16149	26
F			1000	40734	4706
G				40211	3375
H				11423	3965
MS2			502	9147	1612
LCS2			TIM 7/26/98 502	6793	500
BLK 98-02	Org	Am	502	21289	35
97L1879-02F			502	21106 (area)	19601 (area)
G	Org		450	18543 (area)	11831 (area)
H				19791 (area)	15054 (area)
MS2			TIM 7/26/98 450	17615 (area)	5610 (area)
BLK soil LCS2				18425	912
			~~TIM 7/26/98~~		
~~BLK soil 98-03 Total~~		~~Am~~	~~592~~	~~15887~~	incorrect spectra
98-03		Pu	592	15887	162
97L1879-02				~~26509 (area)~~ 29535	~~45957 (area)~~ 47687 TIM 8/10/98
				~~37505 (area)~~ 38360	~~82559 (area)~~ 83599 TIM 8/10/98
BLK LCS3				11856 (area)	7554 (area)
BLK 98-02 Residual		Am	590	26324	5352
97L1879-02F				20724 (area)	13359 (area)
G				23946 (area)	16123 (area)
H				27393 (area)	14929 (area)
97L1879-02 MS2 Residual		Am	926	40388 (area)	10206 (area)
BLK LCS2				43195	153
BLK 98-03 Total		Am	3669	9805	27
97L1879-02				~~21237 (area)~~ 22115	~~4556 (area)~~ 4979 TIM 8/10/98
				~~26019 (area)~~ 26750	~~5871 (area)~~ 6271
BLK LCS3				9108 (area)	3639 (area)

7/30	Sample	Element	Ct Time	Tracer Counts	Analyte Counts
	~~Blk-14~~ NR04	Pu	4000	144952	1152
	~~NR04~~ Blk-14	Pu		137508	145
7/31/98	~~NR04 Blk-14~~ NR04	Am		180232	832
	~~NR04~~ Blk-14	Am		170355	455

8/10/98	Sample	Element	Ct Time 1000m	Tracer Cts	Analyte Cts	
	N002	Am	1000m	5611	317	
	NR02	Am	1000m	4322	48	
	NR023	Am	1000m	5269	148	0.36

9/18/98	Sample	Element	Ct Time (m)	Tracer Cts	Analyte Cts	
	Elec. Sm (NR04)	Pu	1500	6630	6711	
	Elec. Lg (NR04)	Pu	1500	8144	3503	
	N003 0.45	Pu	1500	7588	7841	
	N003 10KMW	Pu	1500	8206	2793	
	Blk-15	Pu	2500	13167	13	
	Elec. Sm (NR04)	Am	1500	566	400	
	Elec. Lg (NR04)	Am	1500	2333	615	
	N003 0.45	Am	1500	2035	574	
	N003 10KMW	Am	1500	1693	158	
	Blk-15	Am	2500	3415	24	

9/18/98

9/21/95 Am-243 spike for efficiency samples

Eff-1 1.0258 g — Am-243 : RH261B
Eff-2 1.0300 g — 6.8869 Bq/g
Eff-3 1.0237 g
Eff-4 1.0250 g

Sample	Det#	Cts	Ct Time	Bq Added
Eff-1	1	137471	1000 m	7.0646
Eff-2	2	136118		7.0935
Eff-3	3	124917		7.0501
Eff-4	4	136075	1000 m	7.0591

Detector Efficiencies (1000 min ct)

Det #	Bq added	cts/s	Eff
1	7.0646	2.2912	0.3243
2	7.0935	2.2686	0.3198
3	7.0501	2.0819	0.2953
4	7.0591	2.2679	0.3213
			$\bar{X}$ = 0.3152

Sample	Det#	Cts	Eff
Eff-2	2	13491	0.3170
Eff-3	3	12930	0.3057
Eff-4	4	13315	0.3144
Eff-1	1	13684	0.3228
			$\bar{X}$ = 0.3150

Detector Efficiencies II (100 min ct)

Det #

9/22/95 TM

9/24/98 QH/QC Sample Data

Sample	Element	Ct Time	fxn	Tracer Cts	Analyte Cts	Tracer Added 13g
Blk 01	Pu	1000m	NA	5955	74	
Blk 01	Am	1000m		3016	22	
Blk-12	Pu	1000m		5819	3	
Blk-12	Am	1000m		3256	10	
Blk 98-01	Pu	1000m	Ox	22321	20	
Blk 98-01	Pu	1000m	Ex	2491	14	
Blk 98-01	Pu	1000m	Carb	2840	7	
Blk 98-01	Pu	1000m	Org	93918	127	
Blk 98-01	Pu	1000m	Res	35031	72	
Blk 98-01	Am	1000m	Org	1637	41	
Blk 98-01	Am	1000m	Res	poor	need	
Blk-13	Pu	1000m	NA	5301	55	
Blk-13	Am	1000m	NA	3147	19	
1-Blk	Pu	1000m	NA	4290	12	
Blk 98-02	Pu	1000m	Ex	5335	574	
Blk 98-02	Am	1000m	Ex	2903	35	
Blk 98-02	Am	1000m	Carb	2964	NA 9	
Blk 98-02	Am	1000m	Ox	16137	26	
Blk 98-02	Am	502 TTM ~~1000m~~	Org	21290	31	
Blk 98-02	Am	590m	Res	26322	53	
Blk 98-03	Pu	592m	Tot	15920	174	
Blk 98-03	Am	366m		9819	31	
Blk 14	Pu	4000m	NA	137698	165	
Blk 14	Am	4000m	NA	170803	510	
Blk 15	Pu	2500m	NA	13170	18	
Blk 15	Am	2500m	NA	3412	26	

TTM 9/24/98

9/25/98 QC Sample Data

Sample	Element	Fxn	Ct Time	Tracer counts	Analyte Cts	Analyte / Tracer By Added
NCS01	Pu	NA	1000m	5632	1049	
SCS01	Pu			115174	3837	
SCS01	Am			140002	790	
NCS01	Am			2981	107	
1-LCS2 TM 9/25/98	Pu			4294	25099	
3-MS1	Pu			~~67552~~	~~8140~~	TM 10/1/98
MS2	Pu	Ex		4399 (area)	163656	
LCS2	Pu	Ex		5455	2432	
LCS2	Am	Ex	1000m	2155 (area)	18698	
MS2	Am	Ex	1000m	poor spectra		
LCS2	Am	Carb	1000m	~~1957 (area)~~ 2373 TM 10/5/98	3292	
MS2	Am	Carb	1000m	1834 (area)	7673	
MS2	Am	Ox	502m	9579	1615	
LCS2	Am	Ox	502m	~~2178 (area)~~ 8818 TM 10/5/98	~~18698~~ 511	
MS2	Am	Org	450m	18250	6035	
LCS2	Am	Org	450m	18457	185	
LCS3	Pu	Tot	592m	1268181	7767	
LCS2	Am	Res	926m	43333	1157	
LCS3	Am	Tot	366m ~~926~~ m	10101	4004	
MS2	Am	Res	926m	41207	10792	

For peaks in which Pu analyte overlaps in the tracer peak, the area given by the analytical software does not appear to be accurate based on a manual extrapolation of the peaks areas. Estimates are therefore given based on the basis of manually (peak tracing, cutting & weighing of the paper mass) determining the overlap.

LCS2 Ex Am gross cts 3185 est % overlap = 12%

- 382 0.12 * 3185 = 382

2803

10/14/98 Extraction Sequence to Obtain Revised Residual Fraction Values for Pu

Samples		grams
97L1879-02	~~K~~ J	3.0
	K	3.0
	L	3.0
	M	3.0
	N	3.0
Sand Blank	16	3.0

Exchangeable Extraction
~16 hours

Carbonate Extraction, eliminated to save time, fraction is small compared to desired residual data

Sesquioxide fraction

Extraction: begin: 3:35 PM 10/14
end: 9:40 PM 11/14

Organic extractions performed as per documented procedure. All of above fractions discarded.

11/4/98

11/4/98 Dilution of ~~SRM~~ Pu-242 Std RH 270

Isotope Products Laboratory Source No: 572-6

Reference Date: Aug. 1, 1997

Initial activity = 98.19 nCi / 5.63826 g = 17.41 nCi/g

Want 100 mL of ~ 300 pCi/g = 0.3 nCi/g

Or 100 g * 0.3 nCi/g = 30 nCi

So: ~~80~~ ~~Dilute entire stock to 50 mL~~

~~for~~ ~ 2 g of stock to 700 mL(g) solution

Original Std transferred to Screw Cap Vial RH 270A	Dilution of Original RH 270B
	gross 30.6928 g
gross 14.4176 g	tare 29.5647 g
tare 8.8176 g	1.1281 g mass of Orig. to container
5.6000 g	
14.4176 g	gross 83.5127 g
13.2887 g (Aliquot taken for dilution)	tare 29.5647 g
1.1289 g	53.9480 total soluti

Specific activity =

$$\frac{1.1281\ g * 17.41\ nCi/g}{53.9480\ g}$$

= 0.364 nCi/g = 364 pCi/g

= 13.47 Bq/g

11/4 Pipettor Calibration

2 M HNO_3

event	mass
1	1.0450 g
2	1.0487 g
3	1.0486 g
4	1.0482 g
5	1.0500 g
6	1.0497 g
7	1.0490 g

$\bar{x} = 1.0485$ g

Dilution of RH 261B

29.0515
tare 28.5354
0.5161

final mass after Dilution 46.4824
tare 28.5354
17.9470

New # RH-261C

Sp. Activity = $\frac{0.5161\text{ g} * 6.8869\text{ Bq/g}}{17.9470}$

$= \boxed{0.1981\text{ Bq/g}}$

11/4/98 TM

11/14/98

Sample	Ct Time	Tracer Cts	Analyte Cts	Tracer Added
02 J Res	60 m	8909	8145	
02 K Res	60	9459	7263	
02 L Res	60	8518	5082	

11/22/98 1 mL (1.0485 g) of RH 270 B added to each sample 02 J-K on 11/4/98.

1 mL (1.0485g) of RH

Sample	Ct Time	Tracer Cts	Analyte Cts	Element	Tracer Added
02 N Res	60	12687	9260		
Blk 98-04	60	12359			
1/21/99 NR06	1000 m	3298		Am	
Blk-17	1000 m	3499	37	Am	