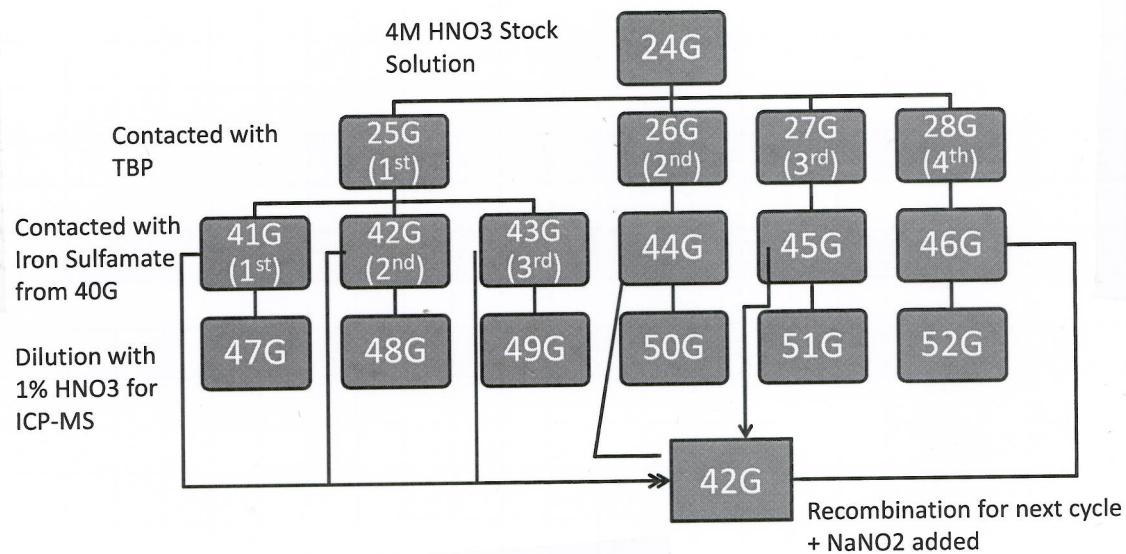


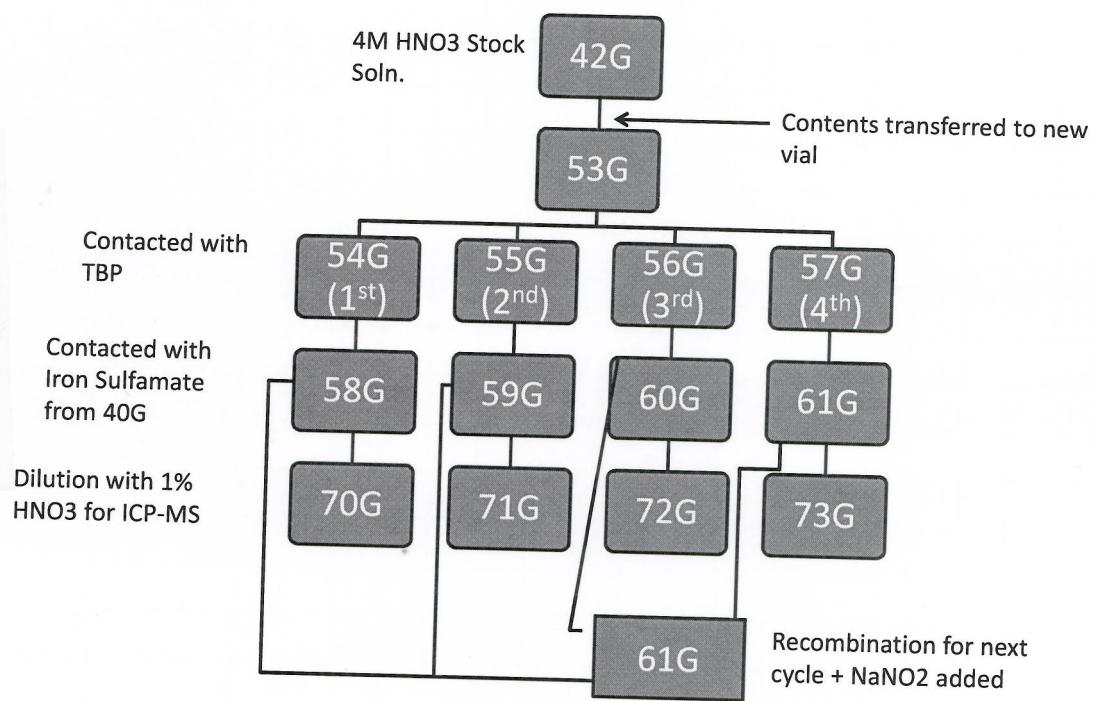
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Paul Cycle 1



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Paul Cycle 2



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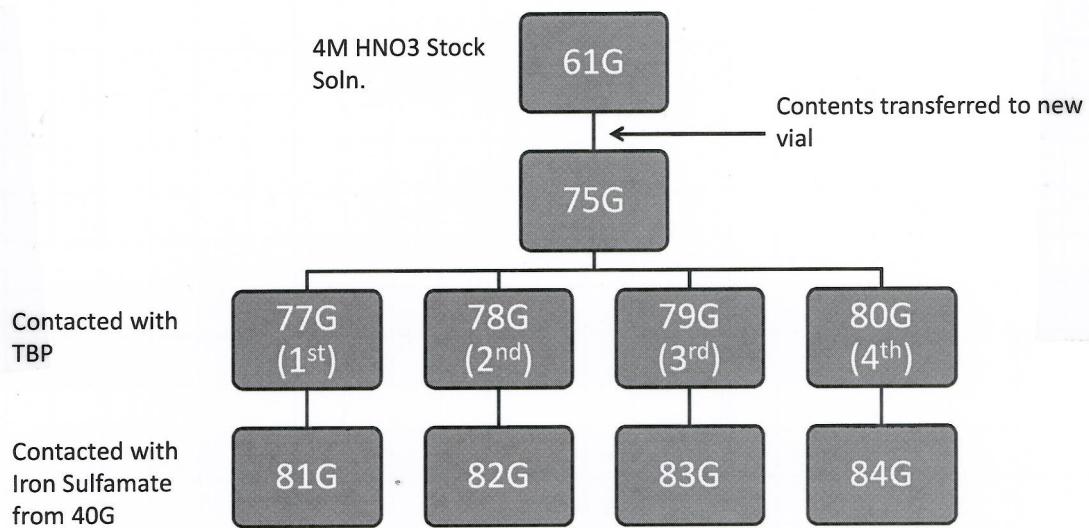
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Paul Cycle 3



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Matt Cycle 1

Mother Vial #	Initial Volume (mL)	Initial Contents	Contact info	Contact #	Volume (mL)	Contact Solution	Transfer Volume approx (mL)	Transfer Contents	Transfer Vial
87G		stock solution (dissolved pellet in HNO ₃ +Kerosene)	4 contacts of 30% Fresh TBP	1	0.7	30% TBP	0.5	TBP+Pu&U	88G
				2	0.5	30% TBP	0.5	TBP+Pu&U	88G
				3	0.5	30% TBP	0.5	TBP+Pu&U	88G
				4	0.5	30% TBP	0.5	TBP+Pu&U	88G
89G	18.263	4M HNO ₃ +0.024M Iron Sulfamate							
88G	2	TBP+Pu&U from 87G	3 FeSO ₄ contacts from 89G	1	2.1	4M HNO ₃ +0.024M Iron Sulfamate (89G)	2	FeSO ₄ +Pu	90G
				2	2.8	4M HNO ₃ +0.024M Iron Sulfamate (89G)	2.02	FeSO ₄ +Pu	90G
				3	2	4M HNO ₃ +0.024M Iron Sulfamate (89G)	2	FeSO ₄ +Pu	90G
90G		FeSO ₄ +Pu from 88G	Sodium Nitrite Contact	1	2	NaNO ₂	0.5	NaNO ₂ + FeSO ₄ +Pu from 88G	91G
91G		Pu+ Sodium Nitrite from 90G	4 contacts of 30% Fresh TBP	1	0.7	30% TBP	0.5	TBP+Pu	92G
				2	0.5	30% TBP	0.5	TBP+Pu	92G
				3	0.5	30% TBP	0.5	TBP+Pu	92G
				4	0.5	30% TBP	0.5	TBP+Pu	92G

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Matt Cycle 1

Mother Vial #	Initial Volume (mL)	Initial Contents	Contact Info	Contact #	Volume (mL)	Contact Solution	Transfer Volume approx (mL)	Transfer Contents	Transfer Vial
92G	2	TBP+Pu from 91G	3 FeSO ₄ contacts	1	2.2	4M HNO ₃ +0.024M Iron Sulfamate	1.99	FeSO ₄ +Pu	93G
				2	2.05	4M HNO ₃ +0.024M Iron Sulfamate	2.01	FeSO ₄ +Pu	93G
				3	2	4M HNO ₃ +0.024M Iron Sulfamate	2	FeSO ₄ +Pu	93G
93G	6	FeSO ₄ +Pu from 92G	Sodium Nitrite Contact	1	2	NaNO ₂	0.5	NaNO ₂ +FeSO ₄ +Pu from 92G	94G
94G	0.5	NaNO ₂ +FeSO ₄ +Pu from 92G	4 contacts of 30% Fresh TBP	1	0.7	30% TBP	0.5	TBP+Pu	95G
				2	0.5	30% TBP	0.5	TBP+Pu	95G
				3	0.5	30% TBP	0.5	TBP+Pu	95G
				4	0.5	30% TBP	0.5	TBP+Pu	95G
95G	2	TBP+Pu from 94G	3 FeSO ₄ contacts	1	2.2	4M HNO ₃ +0.024M Iron Sulfamate (89G)	2	FeSO ₄ +Pu	96G
				2	2	4M HNO ₃ +0.024M Iron Sulfamate (89G)	2	FeSO ₄ +Pu	96G
				3	2	4M HNO ₃ +0.024M Iron Sulfamate (89G)	2	FeSO ₄ +Pu	96G

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10/26

Note: Folder has a class using HPGe on Tues & Thurs.
From 10am → 12pm

Calibration with Eu-152 used (make sure to log source)

10/27

Counted 24G for 2hrs

recorded 12.38% dead time

10/28 Track Down Cs-137

count 25, 26, 27, 28 G
+ potentially further down chain

Ensure clean lab - Make sure Troy has lab moppers

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11/3

Counted the following vials for 1 hr.
after calibrating with Eu-152 (aq.) source for 30 min.

256
266
276
286
296

Summation of Cs-137 Activity found:
1.1614E-2 nCi w/ 5.411% error

this is a 0.0908% of the total
Cs-137 activity

11/4 Meeting

Final's decontamination factor for 1 cycle

(306 trace original - 306 trace)

$$\frac{Ru_{\text{original}} - Ru_{\text{trace}}}{Ru_{\text{original}}} \approx 7$$

Forensics values

Burn up

+ some since irradiation

Deliverables

PUREX - AF , Forensics

Pu purification , gamma analysis + ICPMS

Vial #	Vial Contents	Time Counted (s)	Channel Start	Channel Stop	Centroid	Centroid Energy (keV)	Peak Area (counts)	Error (%)	Activity (dis/s)	Activity (nCi)
24	Contacted Stock Solution (initially 0.5mL)	7200	n/a	n/a	n/a	None at 662	None at 662	n/a	n/a	n/a
25	TBP Contact 1	3600	1804	1813	1808	661.6	213	9.14	0.0592	1.599E-03
26	TBP Contact 2	3600	1803	1813	1808	661.6	735	4.21	0.2042	5.518E-03
27	TBP Contact 3	3600	1804	1812	1808	661.5	299	7.64	0.0831	2.245E-03
28	TBP Contact 4	3600	1804	1814	1809	661.8	300	8.29	0.0833	2.252E-03
29	0.5 mL Stock Solution	3600	1800	1818	1808	661.6	1703911	0.08	473.3086	1.279E+01
25	TBP Contact 1	28800	1804	1813	1809	661.7	1803	3.23	0.0626	1.692E-03

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11/5

Preliminary Procedure
 Leach vials to be used with 3% nitric acid overnight.
 Take aliquot of 4M HNO_3 + dissolved pellet stock soln
 (0.5mL) vial ①

* Count vial ① for 1.5 hrs after performing a background count

Create a 30% by volume TBP soln., diluting with Kerosene
 vial ② mix solution well 21mL kerosene + 4mL TBP

Add Na_2O_2 (0.5mg) to vial ① stock soln (wait overnight)
 converting Pu(III) \rightarrow Pu(IV) via oxidation
 & Pu(VI) \rightarrow Pu(IV) via reduction

Contact vial ① with the 30% TBP from vial ② (0.5mL)
 mix for 15min @ 1500 rpm

* Remove TBP organic phase from vial ① to vial ③
 count vials ① and ③ for 1.5 hrs

Scrub vial ③ with dilute HNO_3 (2M ?)
 mix for 15min @ 15 rpm (use same as TBP dm)

Remove TBP organic Phase from vial ③ to vial ④

* Count vial ③ + ④
 vial 3 + 4 1st Quality Pu

(?) Adjust molarity of vial ④ back to 4M HNO_3

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11/6

Chemicals to be used

4M HNO_3	+ dissolved pellet stock solution	✓ in glovebox lead pty
100% TBP	✓ in acid cabinet under fume hood	
15.44 M HNO_3	Flammable ✓ in acid cabinet	
2.302 M $Fe(NH_4SO_3)_2$	✓ in glovebox	
$NaNO_2$	N_2O_4 powder	✓ in glovebox
	D.I. water	✓ in lab
	Kerosene	✓ in Flammable cabinet

Dilution formula

$$M_{\text{dilution}} V_{\text{dilution}} = M_{\text{stock}} V_{\text{stock}}$$

$$[M] [mL] = [M] [mL]$$

Important

- Always add acid to water
 Leach plastic vials with 5% b.v. HNO_3 overnight;
 Let Na_2O_4 set in vial overnight to allow for complex
 Mix at 1500rpm for 15min each time
 Count after each separation

Will need 50 mL of 0.024 M $Fe(NH_4SO_3)_2$ + 0.75 M

using $\frac{(15.44 \text{ M}) (x)}{50 \text{ mL}} = 0.75 \text{ M } HNO_3$
 $x = 2.429 \text{ mL of } 15.44 \text{ M } HNO_3$

$\frac{(2.302 \text{ M}) (x)}{50 \text{ mL}} = 0.024 \text{ M } Fe(NH_4SO_3)_2$
 $x = 0.521 \text{ mL of } 2.302 \text{ M } Fe(NH_4SO_3)_2$

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DI water needed

$$50 \text{ mL} = 2.429 \text{ mL} + 0.521 \text{ mL} + x$$
$$x = 47.05 \text{ mL DI water}$$

11/9

During Meeting
Ask to see chem for 1 Fe molarity~~HgO~~
N₂O₄ ammonium

Define all reactions of importance taking place

Goal?

Simulate industrial process as closely as possible
Separate out Pd

See Pigford book from Dr. Sunil.

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10/13

Leaching plastic vials to be used 3% HNO_3

$$6 \times 15\text{mL} + 4 \times 45\text{mL} = \\ 90 + 180 = 270$$

$$\frac{300\text{mL}}{9\text{mL } HNO_3} = 286.57$$

#	Size	V(mL)	Use		
1	45mL	30mL	50% TBP Soln	21mL kerosene + 9mL TBP	13.53 15.47M HNO_3
2	45mL	25mL	$Fe(NH_4)_2SO_4$ Soln		
3	45mL	30mL	2M HNO_3	1.2145mL 0.2605mL 23.525mL	15.47M HNO_3 2.302M Fe^{+2} DI water

$$\frac{(15.44\text{M})(x)}{30\text{mL}} = 2\text{M}$$

4	15mL	Dissolved pellet soln	$x = 3.886 \text{ mL } 15.44\text{M } HNO_3$
5	15mL	organic phase 1	$26.117 \text{ mL } DI \text{ water}$
6	15mL	organic phase 2	
7	45mL	30 mL 4M HNO_3	$\frac{15.44\text{M}(x)}{30\text{mL}} = 4\text{M}$ $x = 7.772 \text{ mL }$ 22.228 mL $DI \text{ water}$

10/16 Removed HNO_3 soln from vials to let them air dry
rinse with DI water
Calibrated detector using Eu-152 aqueous source

$$A = 497.0 \text{ nCi}$$

$$t_{1/2} = 13.516 \text{ years}$$

Brought (5H) into glovebox

Removed 0.5 mL aliquot from glass vial stock solution
and moved it into (5H).

Parafilm wrapped (5H) and cleaned surfaces with
radical wash

Took background count before counting (5H) for t hr. 30 min
Placed (5H) back in glove box
Added $NaNO_2$ (0.5 mg)
Mixed at 1500 rpm for 15 min

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11/17 Created stock solutions

1 H	2M HNO_3
2 H	w/o $Fe(NH_4)_2SO_4$ (will add in glovebox)
3 H	50% TBP
4 H	4M HNO_3

Removed 5H from glovebox
 counted 5H again with efficiency calibration
 Replaced 5H back into glovebox

11/18 Generate clear flow -sheet

11/14 Finished generating $Fe(NH_4)_2SO_4$ solution of Fe in (2H) needed to be added in the argon atm.

Re-agitated the (5H) soln.
 Added the 0.5mL of (3H) (50% TBP) into (5H)

~~Shook~~ Mixed the (5H) vial for 15min @ 1500 rpm

Waited 3 min for phases to separate and bubbles to pop

Removed the ~0.5 ml of organic phase off the top and transferred it into (6H)

Sealed (5H) & (6H) with parafilm to transport to counting on HPGe

Counted (6H) for 1 hr

Counted (5H) for 1 hr

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Added 0.5mL from (2H) into (6H) + mixed for 15min @ 1500 rpm
Counted (6H) for 1.5 hrs * bubble in micro pipette led to some aqueous phase being transferred

Counted (7H) for 1.5 hrs

Moved (6H) back to glovebox

Added 0.5mL of 4M HNO₃ to (6H)

Mixed for 15min @ 1500 rpm

Added ~0.5mg of NaNO₂

Mixed for 15min @ 1500 rpm

Let (6H) set overnight for complete oxidation

11/23 glovebox atm reading

<20.0 ppm H₂O + O
P = -1.5 mbar

Mixed (3H) briefly (<1min)

Took pic of (6H)

Added 0.5mL of (3H) into (6H)

Mixed (6H) @ 1500 rpm for 15min

let set for ~5min

Removed ~0.5mL of organic phase of (6H) and transferred it to (8H)

Done in one pull of the micro pipette will lead to do smaller increments for more accuracy in next experiment

* need to refill HPGe with LN₂

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11/23 Chris Crouch # : mobile: 979-820-4105
work: 474-458-1061

11/30 Chris fired, need to find someone to refill
HPGe with liquid nitrogen

HPGe

$$\text{Channels} = 8192$$

$$\text{Max E} = 3 \text{ MeV}$$

$$\text{E calibration} = -5.449 \times 10^{-1} \text{ Rev} + 3.662 \times 10^{-1} * (\text{Ch #})$$

$$\text{Ch \# 1808} = 661.7 \text{ Rev}$$

could use U-235 saw small peak

12/2 ~~12/1~~ Coordinate with Matt Soper for liquid nitrogen
will have ~~12/1~~ 12/3 due to bad hose connection

12/3 Liquid nitrogen delivered

12/7 - Liquid nitrogen has cooled detector
running calibration count (30 min) with aqueous
Eu-152 source.

- Counting (6H)

- Counting (8H)

12/8 Contact ~~(2H)~~ (8H) with 6.5 mL of $\text{Fe}(\text{NH}_4\text{SO}_4)_2$ sol

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Mixed (8H) on vortex mixer for 15 min @ 1500 rpm

removed organic phase from (8H) \rightarrow (9H) after ca 2 min
wait for bubbles to subside

~~recalibrated detector after~~

Counted (8H) for 1.5 hr

Counted (9H) "

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12/16

Added ~0.5mg of Na₂O₂ to (8H)

Mixed (8H) on vortex mixer for 15 min @ 1500 rpm

Let 8H sit overnight

after adding Na₂N₂O₄ soln became red/yellow (oversaturation)added 1 mL 4M HNO₃ + mixed again & color became clear again

12/18

Contacted (8H) w/ 0.5 mL of TBP from (3H)

mixed (8H) for 15 min @ 1500 rpm

removed organic phase ~ 0.5 mL to (10H)

Counted (8H) for 1.5 hr

Counted (10H) for 1.5 hr

Contacted (10H) with 0.5 mL Fe + HNO₃ from (2H)

mixed (10H) for 15 min @ 1500 rpm

Removed organic phase ~ 0.5 mL to (11H)

Counted (10H) for 6 hrs

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12/21 Counted IT H for 4 hrs

1/9 No LN_2 in big tank
Fume hood alarm on (no flow)
Purging glove box
- order 3 tanks (on last one)

Project: 13-075
Phone: (603) 773-9333

E07 error: Box pressure sensor defective
E06 Alarm low pressure in box

3 argon tanks ordered

drain vial - activity sticking? count vials

1/17 adjusted the activity to a per ml unit.
this reduced the error

re-calculated decontamination factor

regenerated glovebox

be in lab at 2pm for video of lab

finishing edit of proposal to sent to other committee members.

in next step will want to count for a longer time
to get better data on ^{29}I Am + ^{29}I In

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Where is the extra activity come from?

weigh pipette volume of water

summation γ -peaks? (move sample farther back)

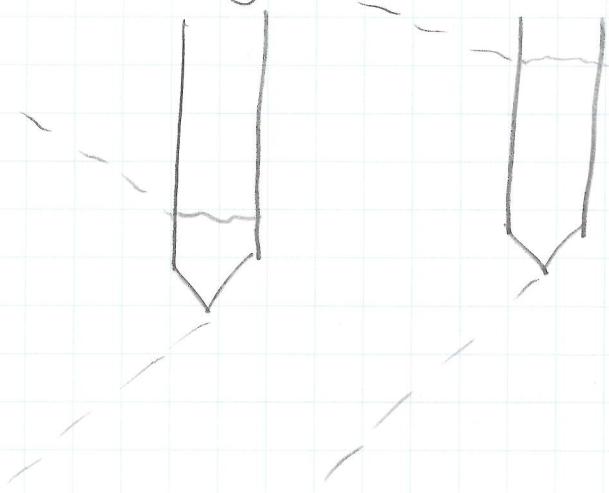
count pipette

creating activity? (talk to Dr. Marland
+ Dr. Foden.)

(Swiney 241-Pu) in his spectra

Find (HF) potentially place in glovebox

Changing Solid angles to detector (dilute small activity sample + count to see difference)



more compton scatter?

1/23 How to reset the HPGe running calibration sample 1 hr

will then run diluted organic sample from (7 H) (9 H)

1/24 7H counted
may need to recount if not enough stirring occurred (11 H)

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2/2 Counted 114 after 5min stir

2/3 Thesis Structure
ensure intro before objectives

this is the work

this is what is expected to occur per literature

this is what occurred

use your own pictures + charts!

use own data to re-enforce

where did the chemicals come from, purified before use?
how are work novel?

Lack of completing ability leaves Alkali metals behind in HNO_3
($n > 6$)

D block easier to be extracted by TBP due to e^- structure

1 of distribution ratio = 50% ; 50% separation (terrible!)

Distribution coefficient

γ_n = gamma count of n phase
solution for certain nuclide

$\frac{\gamma_0}{\gamma_{\text{aq}}}$ = Distrib. Coeff.

γ_{aq}

Answer (for next meeting)

total ~~rate~~ count rate? (each volume)

at different distance same volume?

(further away 1.0ml sample)

Replies after chemistry?

For fast reactor fuel must dilute Pu to 1% for percent to C
or else 3rd layer between organic + inorganic occurs

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Moody book photoca plant example

2/4 Determine total count rate

for

7H

9H

11H

at various volumes + distances

Find more lead bricks for longer distance count
(talk to NSC people)

2/5 Revise proposal + have members of committee sign it

Clamped hose tighter on glovebox gas (working) line
(fixed high H₂O levels)

2/8 Use excel and tabulate count rate data
work on writing methodology

2/9 Begin creating new solutions for round 2 of separation experiments.

Ensure equal volumes when counting

2/10 Which fission product?

for each isotope count rate of photopeak
+ counts

activity of each isotope using photopeak (from efficiency curve of Eu-
activity concentration)

Bring sample up higher so centered to detector

NAS senses on radiochemical science
(national academy of science)

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Web of Science (forward reference) for newer articles that reference .com if.

2/15 Begin Round 2 repetition of process maintain constant volume separations: (Experiment I)

Materials Used

VWR 50mL centrifuge tubes w/ screw caps
disposable / graduated / conical # 21008-189

VWR 15 mL presterilized centrifuge tubes # 09004-361

Water done for 2I + 4I

HNO₃ done for 2I + 4I
TBP + Kerosene done for 3I

2/17 March 15 (Thesis Due)

March 9 Marlene Defense 304 AI 1pm

"

3/7 LN₂ needs to be refilled An HPGe
Lead Bricks set up for longer count distance
Glovebox needs to be maintained

Submission of thesis summer semester?

3/9 Recalibrated HPGe to 8cm v
working on 5cm

Glovebox O₂ + H₂O levels are good for off.
circulation on now.

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3/10

Power outage (Restart detector)
 Calibrated HPLC to 32 cm

When its time to back extract add Fe before to 2I

3/11

Began experiment I

Completed steps 5 → 10

remember to add Fe to 2I before using

Count each pipette tip!

1 mL volume in 5I currently at step 10

3/14 Step 11 Contacted now (5I) w/ 1.0 mL TBA from (3I) 1 min mix before for 3I
 added in 2 ~~from~~ 500 mL increments
 new tip each time

Fe (II) added to (2I)

Step 13 Removed 250 μ L at a time from (5I) to (6I)
 4 times for ~1 mL into (6I)

before step 15 Counted (6I) ~ 13.5 hrs (overnight)

step 14 Counted (5I) ~ 8 hrs

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3/15 Contacted (6I) w/ 1.0mL of (2I) Fe(II).
 Shook mixed (6I) for 15 min @ 1500 rpm
 Let (6I) sit for 5 min

Removed organic phase by 250 μ L x 4 increments
 from (6I) to (7I)

step 19 Counted (6I) ~ 8 hrs

step 20 Counted (7I) ~ 17.5 hrs (overnight)

3/16 Added 0.5 mg NaNO₂ to (6I) + mixed for 15 min @ 1500 rpm
 (let oxidize overnight)

Contacted (6I) w/ 1.0mL of TBP from (3I)
 (in 500 μ L increments)
 new tip each time

mixed for 15 min @ 1500 rpm
 Let settle for 15 min

Removed 250 μ L x 4 at a time from (6I) to (8I)

step 28 Counted (6I) ~ 8.5 hrs

step 29 Counted (8I) ~ 7.1 hrs

3/17 Contacted (8I) w/ 1.0mL of (2I) Fe(II).
 (1 min mix before for (2I))
 Mixed for 15 min @ 1500 rpm

Waited 15 min to settle

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3/17 cont. Removed 250 μ L x 4 from (8I) into (9I)

step 33 Counted (8I) \sim 15.9 hrs (overnight)

step 34 Counted (9I) \sim 13.9 hrs

3/18 Added 0.5 mg of NaNO₃ to (8I)

Let sit from 2:30pm till 10pm

Contacted (8I) w/ 1.0mL of TBP from (3D)
(1 min mix before for

(8I) Mixed for 15 min @ 1500 rpm

Let ~~settle~~ sit for 15 min

Organic removed 250 μ L x 4 from (8I) to (10I)

step 41 Counted (8I) h. \sim 11.3 hrs

Step 42 Counted (10I) \sim 20.67 hrs (overnight)

3/19 Contacted (10I) w/ 1.0mL Fe(II) soln from (2D)
(1 min mix for 22 min)
Mixed (10I) for 15 min @ 1500 rpm

Let set for 15 min

Removed organic from (10I) 250 μ L x 4 to (11I)

3/20 Step 46 Counted (10I) \sim 23.4 hrs

3/22 Step 47 Counted (11I) \sim 24 hrs

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3/23

Group meeting

Need to centrifuge samples after mixing!
~15 min @ 3000 rpm

Asked more stock soln for next exp.

Troy has ~~stock~~ room keys to get source
source
will need 500µL of dissolution soln

3/24

Retrieved dissolution soln + placed new aliquot
into stock soln vial.
(500µL)

Replaced cap of dissolution soln vial
(cap was disintegrating)

Diluted new aliquot to 5mL with 4M HNO₃
from 71

New experiment 5 to begin using centrifuging

Centrifuge info

manuf: Ample Scientific
model: Champion F-33D
Speed to use: 3000 rpm
time: 15 min

Will need ballast vial on opposite side from
sample

Cut more parafilm for glovebox

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Took $500\text{ }\mu\text{l}$ aliquot of stock soln & transferred it to (5J)

Added $500\text{ }\mu\text{l}$ of (4I) HNO_3 to (5J)

Mixed @ 1500 rpm for 5 min

Counted (5J)

Argon Gas Ordered (in afternoon tomorrow)
 3 Argon
 1 Argon / Hydrogen Mix

D - 1.5 mev

$$4046 = 750 \text{ keV}$$

Ch #

8192

E

1,500 keV

4096

750 keV

$$0.183 \frac{\text{keV}}{\text{ch}} \times \text{ch} = \text{keV}$$

$$\frac{14418}{50977} \text{ C} = \frac{28.58}{100}$$

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