Laboratory Journal

Paul Mendoza

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This notebook begins 6 October 2016

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Thursday, 6 October 2016 8:30am - 11:00 am 1:30pm - 3:30pm

1 Isotopes we are looking for

- Decay Monitors
 - ${}^{137}\mathrm{Cs}/{}^{133}\mathrm{Cs}$
- Burnup Monitor
 - $(^{154}\text{Eu}/^{153}\text{Eu}) [^{155}\text{Eu}]$
- Reactor type monitors
 - $(^{134}Cs/^{137}Cs)$
 - $-(^{150}\mathrm{Sm}/^{149}\mathrm{Sm})$
 - $-(^{242}Pu/^{239}Pu)$
 - $(^{135}Cs/^{137}Cs)$
 - $(^{136}Ba/^{138}Ba)$
- Isotope Solve list

$^{136}\mathrm{Ba}$	$^{153}\mathrm{Eu}$
138 Ba	$^{154}\mathrm{Eu}$
$^{149}\mathrm{Sm}$	$^{239}\mathrm{Pu}$
$^{150}\mathrm{Sm}$	$^{242}\mathrm{Pu}$
	$^{138}{\rm Ba}$ $^{149}{\rm Sm}$

Isotope solve list.

2 Experiment Notes

• Project Number: 504370-0001

• EHS Contact:

- d-imenchaca@tamu.edu
- -979-676-0590 For Dan
- 979-845-2132:General
- Files on computer saved in C:/Paul_Mendoza
- ¹⁵²Eu Liquid calibration source
 - Source 1577-22
 - $-497.0~{\rm nCi}$
 - Assy Date: 15 Feb 12
 - -1.00568g
- Stock HNO₃: Assuming Temp= $24.8+/-3 \rightarrow \boxed{Stock\ HNO_3}$
 - Molarity: 15.35 + /-0.13
 - pH: -1.186+/-0.004
 - Molality: 35.3+/-0.8
 - Wt Concentration: 69.0+/-0.5
 - Molar Mass: 63.0130+/-0.0012
 - Density: 1.402 + /-0.006
- Stock Iron Sulfamate $Fe(NH_2SO_3)_2 \rightarrow \boxed{Stock \ Fe(II)}$
 - Molarity: 2.302+/-0.009
 - Molality: 2.717+/-0.006
 - Wt Concentration : 40.26+/-0.05
 - Molar Mass: 248.022+/-0.017
 - Density: 1.418 + /-0.005

3 Stock creation

- Get stock solution from Troy room 18A, store near rad waste
- Grab 1000μ l pipett from glovebox
- Decontaminate with radic dump waste into glass aq rad outside glove box
- Practice pipetting 500μ l to glass vial setting $503~\mu$ l gives $500~\mu$ l
- Class/lunch Break
- Get alpha detector from Dr. Marianno

- Set up laboratory notebook
- Calculation To do calculation to determine the volumes needed for a final concentration of a particular volume, knowing the initial concentrations

$$V_2 = \frac{b_2 - \frac{M_1 b_1}{A}}{M_2 - \frac{M_1}{A}}$$
$$V_1 = \frac{b - BV_2}{A}$$

Where:

$$A = (1 - wt\%_1)\rho_1$$

$$B = (1 - wt\%_2)\rho_2$$

$$b_1 = (1 - wt\%_3)V_3\rho_3$$

$$b_2 = M_3V_3$$

With known Molarity and volume of a solution how much, and of what concentration do we need to combine with a second solution to get a final solution of known concentration and volume?

$$B = (1 - wt\%_3)V_3\rho_3 - (1 - wt\%_1)V_1\rho_!$$

$$A = M_3V_3 - M_1V_1$$

$$C = \frac{B}{A} = \frac{(1 - wt\%_2)\rho_2}{M_2}$$

Need iterative solution, choose:

$$M_2 = \frac{M_3 V_3 - M_1 V_1}{V_3 - V_1}$$
$$V_2 = V_3 - V_1$$

Use to determine molality $\to wt\%_2 \to \rho_2$. Then compare to C, iterate around the solution to find answer so that $C = \frac{(1-wt\%_2)\rho_2)}{M_2}$.

Friday, 7 October 2016 9:00am - 12:00 am 1:00pm - 4:00pm

1 Stock creation

- ✓ Program calculation for creation of stock some results shown below
- - Clean off and move leaded shielding in rad area to countertop next to fume-hood
 - Add diaper paper on countertop, and on shielding incase of contamination
 - Practice transfer

√ _

$$0.149+/\text{-}0.011 \text{ ml of } 15.43+/\text{-}0.06 \text{ M HNO}_3 \boxed{Stock\ HNO_3} \\ + \\ 1.91+/\text{-}0.08 \text{ ml of } 0.0+/\text{-}0 \text{ M solution } \boxed{DI\ Water} \\ = \\ 2.048+/\text{-}0.026 \text{ ml of } 1.12+/\text{-}0.08 \text{ M HNO}_3 \text{ solution } \boxed{\rightarrow Stock} \text{ (glass container)}$$

✓ -

- ✓ Put Source back in rad closet
- ☑ Clean up contamination added to pipette tip from transfer (for some reason, the contamination was added to the inside of the pipette itself, the tips used don't have the block, but still, none of the solution should have traveled up the shaft

- ☑ Dispose of diaper paper laid down for transfer (where the glass bottle was set down which contained closet solution, there was contamination (the outside of the bottle of the closet solution is contaminated)
- ✓ Move shielding back to where it was

2 Preparation for Process 1

- ☑ Count calibration standard Eu-152 in HPGe 3 hours 22 minutes at furtherest position from detector (26 cm)
 - Source 1577-22
 - 497.0 nCi
 - Assy Date: 15 Feb 12
 - 1.00568g
- Create Eu-152 Excel Counting sheet template for standards
- 🗹 Set up ROI (region of interest) file for Eu-152
- - Count lasted for 12 hours

Saturday, 8 October 2016 10:00am - 2:00 pm

✓ Finish background count, lasted 12 hours

1 Preparation for Process 1

centrifuging

- ✓ Remove 0.3 ml from Stock transfer to 1 for counting
 1 is a smaller tube, which will fit into a larger centrifuge tube for, well,
 - 1 tube cannot fit into centrifuge tube with white push cap (pushes on outside of tube), white push cap is necessary when votex mixing, so a blue push cap (pushes on inside of tube), was put on for counting, these smaller tubes will have to have two caps following them around, I can't wait till the second cycle when the bigger tubes will be used
 - \bullet Note for why smaller tubes are being used: when pipetting the smaller volume of 0.3 ml for aq/o phase separation it is much easier to have the smaller diameter tubes
 - Stock was removed from glovebox, and after was put into the safe
- Fix density calculation in code, was slightly wrong before, this means Stock and are slightly different from what they should be, but within error
- ✓ Calculation for creation of Fe(II) solution (next page)

$$V_1$$
 ml of $M_{1,Fe}$ Fe(II) in M_{1,HNO_3} HNO₃ + V_2 ml of $M_{2,Fe}$ Fe(II) in M_{2,HNO_3} HNO₃ = V_3 ml of $M_{3,Fe}$ Fe(II) in M_{3,HNO_3} HNO₃.

The knowns are:

$$M_{1,Fe}=2.302,~\rho_1=1.418,~M_{1,HNO_3}=0$$
 (Fe Stock soltuion) $M_{2,Fe}=0, \rho_2=\rho_{HNO_3}(M_{2,HNO_3})$ $V_3=4$ ml, $M_{3,Fe}=0.024,~M_{3,HNO_3}=4,~\rho_3=\rho_{HNO_3}(4M)$

Mols of Fe(II) constant:
$$V_1=\frac{M_{3,Fe}V_3}{M_{1,Fe}}=0.042$$

Mols of HNO₃ constant: $V_2=\frac{V_3M_{3,HNO_3}}{M_{2,HNO_3}}$
Mass Constant: $V_2=\frac{V_3\rho_3-V_1\rho_1}{\rho_2}$

Combine last two equations:
$$M_{2,HNO_3} - \frac{V_3 M_{3,HNO_3} \rho_2}{V_3 \rho_3 - V_1 \rho_1} = 0$$

Solve iteratively (where M_{2,HNO_3} determines ρ_2) with first guess of: $M_{2,HNO_3} = \frac{M_{3,HNO_3}V_3}{V_2}$

Sunday, 9 October 2016 7:30 pm - 11:30 pm

1 Preparation for Process 1

✓ Prepare for multi contact extraction and back extraction exp

- Make solution of 30 vol.% TBP with kerosene
- Make 40 ml of solution 4.06 M HNO₃ solution,
- Transfer two smaller vials (one for TBP phase), one for Fe phase, with two different lids into glovebox (with a larger vial to hold them in the centrifuge)
- Transfer two smaller vials with centrifuge vials for centrifuging, keep one with water 0.3 ml, and TBP mix 0.32 ml $\boxed{Vial~1~Budd}$, and the second with 1.2 ml of TBP mix and 1.25 ml water $\boxed{Vial~2~Budd}$
- Transfer Stock and $\boxed{1}$ to glovebox
- Transfer another vial to hold the Fe solution
- Make sure tweezers are in glovebox (they are) to remove smaller vials from centrifuge tubes
- Transfer slightly contaminated pipette to glovebox
- All above vials that would contain solution were rinsed with whatever they would hold for approximately 3 minutes

1 _

15+/-0.15 ml of TBP
$$\boxed{Stock\ TBP}$$
 + 35+/-0.35 ml of kerosene $\boxed{Stock\ kerosene}$ = 50+/-0.5 ml of 30 vol.% TBP. $\boxed{\rightarrow TBP}$

✓ _

$$10.579+/\text{-}0.011 \text{ ml of } 15.35+/\text{-}0.13 \text{ M HNO}_3 \boxed{Stock \ HNO_3} \\ + \\ 30.355+/\text{-}0.030 \text{ ml of } 0.0+/\text{-}0 \text{ M HNO}_3 \text{ solution } \boxed{DI \ Water} \\ = \\ 39.94+/\text{-}0.14 \text{ ml of } 4.07+/\text{-}0.04 \text{ M HNO}_3 \text{ solution } \boxed{\rightarrow Fe \ Prep}$$

To create an Fe solution for a back extraction, $Fe\ Prep$ should be combined in the following manner (Small portions created because this solution has a short half life with larger concentrations of HNO_3).

_ -

$$\begin{array}{c} 0.0417 + /\text{-}0.0018 \text{ ml of } 2.302 + /\text{-}0.009 \text{ M Fe(II) in } 0.0 + /\text{-}0 \text{ M HNO}_3 \\ & + \\ 3.941 + /\text{-}0.027 \text{ ml of } 0.0 + /\text{-}0 \text{ M Fe(II) in } 4.06 + /\text{-}0.05 \text{ M HNO}_3 \text{ solution } \\ & + \\ 4.000 + /\text{-}0.020 \text{ ml of } 0.0240 + /\text{-}0.0010 \text{ M Fe(II) in } 4.00 + /\text{-}0.05 \text{ M HNO}_3 \text{ solution } \\ & - \rightarrow Bk \ Ex \ Solution \end{array}$$

- Add Sodium Nitrite to 1, it will sit overnight, but it doesn't have to
 - Dropped 1, solution probably contaminated blue lid (crap), centrifuged on 1000 rpm for 2 minutes

Monday, 10 October 2016 12:30 pm - 4:30 pm

1 Process 1 Mistake experiment

✓ First contact - Extraction

- Add $0.32 \text{ ml } \boxed{TBP} \text{ to } \boxed{1}$
- Shake on Pulse Mode of 15 minutes on vortex mixer
- Change of plans (This occurred while sample settled for a bit while changes were implemented)
 - Put smaller tubes directly into centrifuge so we do not have to switch caps so often
 - Pulled out Vial 1 Budd and Vial 2 Budd Pulled out of glovebox the smaller tubes, changed their caps, labeled them, put back into glovebox (5-10 minutes)
- Centrifuge 1000 rpm for 10 minutes
- Attempted to pull out 0.30 ml of TBP phase
 - Utter Failure
 - Utter Failure again
 - Utter failure...difficult to pull out 0.3 ml and keep phases separate
- Added 1.08 ml \overline{TBP} to $\boxed{1}$ (for 0.2 ml buffer)
 - All extractions at once (different from original exp)

$$p = \frac{1}{1 + \frac{1}{D} \frac{V_{aq}}{V_a}}$$

- $-V_o$ increased by fourfold
- Pipette slipped to 538 (instead of $540 \rightarrow 0.4\%$ increase in error)
- Vortex mix for 15 minutes on pulse mode
- Centrifuge 1000 cpm for 10 minutes
- Remove 1000 ml top phase (TBP), then remove another 200 ml of top phase (TBP) $\rightarrow 2$

 $0.0417 + /-0.0018 \text{ ml of } 2.302 + /-0.009 \text{ M Fe(II) in } 0.0 + /-0 \text{ M HNO}_3$ $Stock\ Fe(II)$

3.941+/-0.027~ml of 0.0+/-0 M Fe(II) in 4.06+/-0.05~M HNO $_3$ solution $\boxed{\textit{Fe Prep}}$

4.000+/-0.020 ml of 0.0240+/-0.0010 M Fe(II) in 4.00+/-0.05 M HNO₃ solution $\rightarrow Bk\ Ex\ Solution$.

- ☑ Back Extraction First Contact
 - Add 1.4 Bk Ex Solution to 2
 - Shake pulse mode for 15 minutes
 - Remove 1.2 ml of bottom phase (Fe(II)) $\rightarrow 3$
 - Lost two drops
 - While placing vial into centrifuge, cap shot off, spraying solution everywhere...great
- ☑ Back Extraction Second Contact
 - Add 1.2 $Bk \ Ex \ Solution$ to $\boxed{2}$
 - Shake pulse mode for 15 minutes
 - Remove 1.2 ml of bottom phase (Fe(II)) $\rightarrow 3$
- - Add 1.2 $Bk \ Ex \ Solution$ to $\boxed{2}$
 - Shake pulse mode for 15 minutes
 - Remove 1.2 ml of bottom phase (Fe(II)) $\rightarrow 3$

This experiment had sputtering of pipette at certain times.

2 Counting for Process 1 Mistake experiment (Gamma)



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Tuesday, 11 October 2016 10:30 pm - 1:00 am

1 Counting for Process 1 Mistake experiment (Gamma)





First Three Counts



Second Three Counts

Wednesday, 12 October 2016 11:30 am - 1:30 pm

1 Counting for Process 1 Mistake experiment (Gamma)

- \square Finish count $\boxed{3P}$
- - Determined ¹³⁷Cs, ¹⁴⁴Ce, ¹⁰⁶Rh activities for first 4 counts Excel sheet
 - Used excel sheet from John Burns for efficiency calibration of Eu-152 source...will just use the sheet from now on
 - Also got from John, a templating file for GENIE, "AnalysisMG.tpi", which helps a lot for output from GENIE, again, something I do not want to modify
 - The template was in an algorithm from GENIE, had the following steps
 - 1. Peak Locate Unidentified 2nd Diff
 - Channels 1-16000
 - -2.50
 - 0.50 FWHM
 - Add to existing results
 - 2. Peak Area Sum/Non-linear LSQ Fit
 - Channels 1-16000
 - 4 channels, use fixed tail parameters
 - Channels, Step, 4.00, 4.00, 4.00
 - Output to screen and printer
 - 3. Reporting...
 - "AnalysisMG.tpi", "C:/GENIE2K/CTLFILES/"
 - PeakAnalysis, 1.000000
 - Start on: Page One, New File, μCi
- ✓ Notes for research meeting
 - Process dilutes by factor of 12, no matter what

- \bullet Concentrated stock by a factor of two
- Decreased initial volume
- Have to maintain, 0.2 ml excess volume to pipette from top
- \bullet Have to maintain, 0.1 ml excess from bottom
- \bullet Mistake in extraction all extractions at once

Thursday, 13 October 2016 12:30 am - 4:30 pm

1 Counting for Process 1 Mistake experiment (Gamma)

✓ Finish count 2W

2 Counting for Process 1 Mistake experiment (Alpha)

- ✓ Start count 2
- ✓ Fix alpha counter, reivew alpha counting
 - Alpha detector broken, fixed by plugging into proper port
 - Counted Calibration Alpha source
 - There are some details for determining what the alpha efficiency should be for the alpha detector, and I want to make sure I do it correctly, have not had time to look into it. I have a PDF file that shows what is in the sample
 - /notebook/Figures/Alpha_Copy.pdf
 - Pu-239 and Pu-240 are unresolved
 - Pu-238 and Am-241 are unresolved
 - Isotope Droduets Laboratories
 - 38.81 nCi
 - -1451-68-3
 - 1 Dec 10
 - Kevin also provided me with a Excel Sheet that does some of the calculations, probably will have to modify
- Counted Alpha Background
- - From Jarrod's stock $10\mu l$ was diluted to 1ml and 10 μl was taken

10
$$\mu$$
l of $Stock$ (4 M HNO₃) + 190 μ l of DI water (leftover in glovebox) 990 μ l of DI water (leftover in glovebox) = 0.2 ml of \sim 0 M HNO₃ 4 Dilution 1 ml of \sim 0 M HNO₃ \rightarrow 4 Dilution

- - Take 20 μ l of 4 Dilution, put onto concentric circle disk plates (innermost circle) $\boxed{D1}$
 - It should be noted that once an alpha source is placed on these disks and dried out, they look no different from other disks
 - Let dry in glovebox

Friday, 14 October 2016 8:30 am - 9:00 pm

Finish count 2

2 Counting for Process 1 Mistake experiment (Alpha)

 $\mathbf{\underline{\checkmark}}$ Finish count for $\boxed{D1}$

3 Analysis for Process 1 Mistake (Gamma)

☐ Attempt to understand our alpha efficiency (basically how much is in the calibration source)

Monday - Wednesday, 17-19 October 2016

1 Analysis for Process 1 Mistake (Gamma)

- \square Looked into alpha calibration math some more
- 🗹 Analyze and automate (somewhat) Gamma analysis
 - Program for pulling peak data from GENIE
 - Program for calculating efficiency from peak energy data using John Burn's Excel file
 - Determine Compton Edges for peaks

$$E_f = \frac{E_i}{1 + \frac{E_i}{511}(1 - \cos\theta)}$$

$$E_i = \frac{E_f}{1 - \frac{E_f}{511}(1 - \cos\theta)}$$

- Found that I do not have any back scatter peaks
- Program for finding sum peaks
 - Included backscatter peaks
 - Found some coincidence peaks, didn't know how to analyze
- Quantify most of the peaks in gamma spectrum (took the longest)

$$CPS = A\gamma\epsilon$$

$$CPS = A_1 \gamma_1 \epsilon_1 + A_2 \gamma_2 \epsilon_2$$

- Most peaks used the first equation, one peak had overlapping energies, so used the second equation, had to assume one of the activities
- Applied this analysis to 6 gamma spectrum (took second longest now more automated)
- Create graphics to help depict what work was actually done
- ✓ Note: Follow these steps when analyzing Gamma

- 1. Make sure Efficiency Excel Sheet is up to date
 - Run Eff Count and particular distance
 - Run: "Analyze Execute Sequence Analyze_Data" on GENIE
 - Save as a .PDF (not .pdf) file the spectra data : File Export Report to PDF from GENIE
 - Pull Peak information with Data_Pull.py program (direct program to directory with .PDF file)
 - Put data into spreed sheet "C:/Rad_Detection/Calibration/Gamma/Eff_cal_summary_Eu-152.xlsm"
- 2. Gather data in a similar manner as with the efficiency count will produce a bunch of plain Excel Sheets
- 3. Find the template from C:/Rad_Dection folder, update real Eff column with "Eff_Calc.py" (Make sure you copy paste energies into the gamma_energies file)
- 4. Copy this template over to the sheets you just made, and gamma analysis for the peaks will be complete
 - Note: Will have to copy, paste, remove peak columns that were not found or in excess from template, lining up everything and then delete was copied over, then paste again, janky, but not super slow - this list is a reminder for Paul, if anyone else is using this list, would probably need more explanation

✓ Notes for Research Meeting

- Showed activities for each of the solutions
- Found that D-values couldn't be found because of experimental setup
- Activity Balance seemed to match up
 - Although it wasn't perfect because the numbers weren't exactly close to zero, but within the error
- Results seemed to match up with previous experiment
- Moving Forward, John and Sunil and I discussed what these next experiments should entail

Thursday, 20 October 2016

1 Preparation for 3 Cycles

Note from John:

After the research meeting yesterday, I thought about Pauls project quite a bit and what the best path forward should be. In my opinion, it would be best for him to do a single-cycle (extraction/back extraction) in a replicate of 3 and determine the D-values for both the extraction and back extraction and show the reproducibility of this single-cycle experiment. I believe this is one of the goal you set for him as a part of his proposal. From there we can move into the whole process with confidence that we have consistent behavior for Cs-137 and Cs-134, as well as, a good understanding of the D-values for the isotopes of interest that can be seen by gamma-ray analysis. He and I spent some time this morning talking about this and we both agree that this week he will focus on completing all 3 single-cycle replicates, gamma counting all the solutions, alpha counting as many as possible (I do not believe alpha and gamma counts cannot be performed at the same time, as they both use the computer), and analyzing a majority of the data before next weeks research meeting. If you do not think this is plan of action in the best to pursue we can restructure it.

I spend the rest of the day doing homework, I aplogize, but it was due yesterday, I think its dumb that I should have to apologize for spending **ANY** time doing homework.

John also mentioned two good techniques, that should be noted:

- Pipetting with equal volumes using the plastic squish tops
 - Squeeze top while going through organic, suck up as much as possible
 - Then draw from top as well
- Measureing volume with pipette
 - The above technique would need some means for measuring volume using the pipette, you can vary the volume around what you thought you sucked up, and check if there is air at the bottom of the tip

Friday, 21 October 2016 9:30am - 12:00 pm 1:00 pm 6:00 pm

✓ Updated this lab notebook (most of this morning)

1 Preparation for 3 Cycles

- Practice pipetting out with squish tops like John Mentioned
 - Used Kerosene solution, used squish pipettes and variable pipettes settled upon using 500 μ l and taking out 350 μ l and then getting as much out as possible with the squish pipette I get about 450 μ l of bottom phase (HNO₃) and 425 μ l of top phase (TBP)
 - Determine if 0.3 ml is a good amount of solution to use
 - Switching to 0.5 ml, keeping smaller vials
- ✓ Create and label vials 5 6 and 7 to hold stock solution. Did not leech them, hopefully barium contamination wont be a huge deal, we will assume all the data for Cs can be gathered from ¹³³Cs.

- \checkmark Transfer 0.5 ml of \boxed{Stock} to $\boxed{7}$
- ✓ Add scoop of sodium nitrite to 6
- ✓ Add scoop of sodium nitrite to 7
- ${f C}$ Centrifuged ${f 5}$, ${f 6}$ and ${f 7}$ to push all solution to botttom of vials
- \square Start count of $\lceil 5 \rceil$ noticed bubbles in solution, might have to recount left overnight

2 Counting for Process 1 Mistake experiment (Alpha)

- ${f {\it v}}$ Took 20 μ l out of ${\bf \bar 3}$ and put onto planchet chip (no dilution)
 - Moved chip too early (before drying, ruined detector volume)
 - Made another source with an additional 20 μ l, letting it dry over night

Saturday, 22 October 2016

3:30 pm - 3:45 pm

8:00 pm - 8:30 pm

1 Preparation for 3 Cycles

- ✓ Finished count for 5
- ✓ Started count of 6
 - Switching from push clear caps to blue push caps
 - This sample had less bubbles than the one yesterday
- ✓ Finished count of 6
 - Some liquid was not at the bottom of the vial, messing with geometry, centrifuged with 7 might have to recount
- ✓ Started count of 7

Sunday, 23 October 2016

1 Preparation for 3 Cycles

- ✓ Finished count 7
- - Did not like how 6 didn't fit with others
- ✓ Started recount of 6

Monday, 24 October 2016 10:00 am - 12:00 pm 3:00 pm - 8:00 pm

1 Preparation for 3 Cycles

- Finished count 6
- ✓ Transfer:
 - Vials labeled $\boxed{5\ Aq}$, $\boxed{5\ Or}$, $\boxed{6\ Aq}$, $\boxed{6\ Or}$, $\boxed{7\ Aq}$, $\boxed{7\ Or}$
 - With clear push lids, and blue push lids (named)
 - Squish pipettes

Into glovebox small antichamber

- [5], [6], and [7] already in antichamber
- ✓ Transfer vials with clear lids into glovebox, but leave the blue lids in the antichamber (lid transfer area)

2 Counting for Process 1 Mistake experiment (Alpha)

- - Saw energy smearing for counts
 - Preliminary results are what was expected if we take a larger range of counts

3 Cycle experiment, replicate of 3

- ☑ Shake 5 on Pulse mode for 15 minutes

☑ Shake 6 on Pulse mode for 15 minutes

 \mathbf{Z} Create EXBuddy so all samples can be centrifuged together

- 500 μ l of 4 M HNO₃ + 500 μ l of 30 vol.% TBP
- ✓ Centrifuge samples for 3000 rpm for 5 minutes
- ✓ Separate phases for samples
 - A total of 4 drops were dropped in this process
 - 1. Sample 5 aqueous transfer
 - 2. Sample 6 organic transfer
 - 3. Sample 7 aqueous and organic transfer
 - Using a variable pipette and the squish pipette, as much of the top phase (organic) phase was removed as possible (turns out to be around 450 μ l and transferred to $\boxed{5~Or}$, $\boxed{6~Or}$, and $\boxed{7~Or}$.
 - Then as much of the bottom phase (aqueous) was removed as possible (turns out to be around 430 μ l) and transferred to $\boxed{5 \ Aq}$, $\boxed{6 \ Aq}$, and $\boxed{7 \ Aq}$.
- ${\bf Z}$ Measure Volumes of 9 vials (Aqueous, organic, and original units of μ l)
 - Clean outside of vials before taking volume measurements
 - Centrifuge vials before taking volume measurements
 - Google says that 1 drop of water is about 50 μ l

Series	Aqueous	Organic	Original	Should Add To	Missing
5	461+/-9.22	430+/-8.6	55+/-5	1000+/-7.1	54+/-15.3
6	469 + / -9.38	430 + / -8.6	53 + / -5	1000 + /-7.1	48+/-15.4
7	469 + / -9.38	430 + / -8.6	57.5 + /-5	1000+/-7.1	43.5+/-15.4

 ${\bf \Box{$\it C$}}$ Count ${\bf \Box{$\it T$}}$ 12:00 pm - 6:00 pm

- - Will try and implement this:

$$CPS = A\epsilon_D\epsilon_G\gamma$$

Where:

$$\epsilon_D = \text{Detector eff}$$
 $\epsilon_G = \text{Geometric eff}$
 $\gamma = \text{yield}$
 $A = \text{activity}$

At two different distances 1 and 2:

$$CPS_1 = A\epsilon_D\epsilon_{G1}\gamma$$
$$CPS_2 = A\epsilon_D\epsilon_{G2}\gamma$$

Take ratio:

$$\frac{CPS_1}{CPS_2} = \frac{A\epsilon_D\epsilon_{G1}\gamma}{A\epsilon_D\epsilon_{G2}\gamma} = \frac{\epsilon_D\epsilon_{G1}}{\epsilon_D\epsilon_{G2}} = R$$

Kept both efficiencies because calibration lumps both together. If This ratio, R is known, then we can count at a closer distance and say:

$$CPS_2 = \frac{CPS_1}{R}$$

 ${\bf extbf{ extit{M}}}$ Move $\boxed{6~Or}$ and $\boxed{7~Aq}$ to Antichamber (not sure which one I am counting next)

4 Calculation Work

- ✓ Modify program for analyzing spectra
 - Hopefully now analyzing gamma data will just be, run program, and copy a part of an excel spreedsheet

Tuesday, 25 October 2016 8:00 am

1 Cycle experiment, replicate of 3

2 Contamination spill 10/25/16

- \Box Go to count $\boxed{5 Or}$
 - Have $\boxed{7 \ Or}$ and $\boxed{7 \ Aq}$ in small antichamber
 - Put antichamber to vacuum to transfer vials into glovebox
 - Push caps exploded off vials due to large pressure difference...that is very dissapointing
- - Dispose of counting vials, and caps for all vials rad waste
 - Dispose of exploded vials in rad waste (after dried)
 - Remove diaper paper from transfer plate
 - Clean with radiac wipes
 - Clean antichamber
 - Clean antichamber
 - Swipe area, count on alpha detector, because our swipe counter is down
 - Clean antichamber
 - Dr. Chirayath brought someone by to talk, not a good time
 - Clean antichamber
 - Clean glass beaker that was in antichamber...lots
 - Final areas swiped and counted for 10 minutes after decontamination
 - Tray ~ 0 counts in alpha realm
 - Top part of cylinder of antichamber ${\sim}3$ counts in alpha realm (around 20 for background)

- Top back part of cylinder ~ 100 still slightly contaminated, but no time for continued cleaning, because need to do experiment
- Left/Right side of cylinder (mid plane) \sim small
- Bottom back portion of cylinder of antichamber ~ 100
- Glass vial none

3 Cycle experiment, replicate of 3

- \square Count $\boxed{7 \text{ } Aq} \boxed{9:00 \text{ pm} 11:00 \text{ pm}}$ (Spilled)
- **Z** Count 6 Aq 7:00 pm 9:00 pm
- \square Count $\boxed{5 \ Aq} \ 9:00 \ \mathrm{pm}$ 8:00 am
- **✓** -

 $0.0417 + /-0.0018 \text{ ml of } 2.302 + /-0.009 \text{ M Fe(II) in } 0.0 + /-0 \text{ M HNO}_3$ $Stock\ Fe(II)$

3.941+/-0.027 ml of 0.0+/-0 M Fe(II) in 4.06+/-0.05 M HNO $_3$ solution $\[Fe\ Prep\]$

4.000+/-0.020 ml of 0.0240+/-0.0010 M Fe(II) in 4.00+/-0.05 M HNO₃ solution $\rightarrow Bk\ Ex\ Solution$.

- \square Add XX μ l Fe(II) solution to $\boxed{7 Or}$ (spilled)

- $\hfill \square$ Shake $\fbox{7~Or}$ 15 minutes on pulse mode (spilled)
- \square Remove XX μ l organic and XX μ l aqueous from Ex Buddy (No longer necessary)

✓ Separate phases for samples

- A total of 1 drops were dropped in this process
 - 1. Sample 5 Or aqueous or organic transfer
- Using a variable pipette and the squish pipette, as much of the bottom phase (aqueous) phase was removed as possible and transferred to $\boxed{5~OrII}$, $\boxed{6~OrII}$, and $\boxed{7~OrII}$.
- Then as much of the top phase (organic) was removed as possible and transfered to $\boxed{5~AqII}$, $\boxed{6~AqII}$, and $\boxed{7~AqII}$.
- \square Measure Volumes of 9 vials (Aqueous, organic, and original units in μ l)

	Series	Aqueous II	Organic II	Original II	Should Add to	Missing
Ī	5	407+/-8.14	380+/-7.6	38+/-5	860+/-12.2	35.0+/-17.2
	6	402415+/-8.3	360380+/-7.6	35+/-5	860+/-12.2	30+/-17.3

4 Calculation Work

☑ Updated Spreedsheets to calculate activities based on available peaks, also if a particular peak has really large errors, this will be ignored. Also updated Excel sheets to calculate propagated error mass in each vial - for D-value calculations

$$grams = \frac{\text{Activity} \times \text{Molar Mass}}{\lambda_s N_A}$$

where λ is in seconds and N_A is avogadros number.

Wednesday, 26 October 2016 8:00 am

1 Cycle experiment, replicate of 3

- \mathbf{Z} Finish count $\boxed{5 Aq}$
- ✓ Start count 6 AqII
- Analyze current spectra
 - Calculate activity (with error) for vials $\boxed{5}$, $\boxed{6}$, $\boxed{7}$, $\boxed{5}$ A, $\boxed{5}$ O, $\boxed{6}$ A, $\boxed{6}$ O,
 - Calculate, for those same vials (with error, even including error on molar mass), mass of each radioactive species, and the concentration (g/L)
 - Compared all first solution activities and concentrations, they were all very similar
 - Compared ¹³⁷Cs ¹³⁴Cs ratio, and they agreed between vials
 - Determined activity balance, making sure each cycle had balance of activity (measured a part of the solution 459/500, found grams per liter, and multiplied by 400).
 - Agreed within the error
 - Determined D-values from aqueous and organic solutions, compared same elements different isotopes
 - The numbers did not look super similar, but sort of similar

$$O\% = \frac{1}{1 + \frac{V_A}{V_O D}} \Rightarrow D_O = \frac{1}{\frac{V_O}{V_A}(\frac{1}{O\%} - 1)}$$

$$A\% = \frac{1}{1 + \frac{V_O D}{V_A}} \Rightarrow D_A = \frac{V_A}{V_O} (\frac{1}{A\%} - 1)$$

Where O and A represent organic and aqueous, where V is volume and % refers to mass percent in a particular phase. The mass percent was determined via:

$$\% = \frac{\text{Mass Part}}{\text{Total Mass}} = \frac{c \left[\frac{g}{L}\right] \cdot V_{\text{contact}}}{\text{Mass in original}}$$

- Propagate error for D-value calculation (as well as for others)
 - Attempted to install uncertainties onto python on windows system, but failed epically, windows is terrible
 - Instead used uncertainties on linux based system to check my answers for the below codes

Aqueous D-value calculation

$$\sigma_{D_A}^2 = \left[\frac{\sigma_{V_A}}{V_O} \left(\frac{1}{A\%} - 1 \right) \right]^2 + \left[\frac{V_A \sigma_{V_O}}{V_O^2} \left(\frac{1}{A\%} - 1 \right) \right]^2 + \left[\frac{V_A \sigma_{A\%}}{V_O A\%^2} \right]^2$$

Organic D-value calculation

$$\sigma_{D_O} = \sqrt{\left[\frac{\sigma_{V_O}}{V_A} \left(\frac{1}{O\%} - 1\right)\right]^2 + \left[\frac{V_O \sigma_{V_A}}{V_A^2} \left(\frac{1}{O\%} - 1\right)\right]^2 + \left[\frac{V_O \sigma_{O\%}}{V_A O\%^2}\right]^2} \cdot D_O^2$$

✓ Create graphic to explain these results to research group

2 Contamination spill 10/25/16

- Treate graphic of all alpha spectra and locations of swipes
- Z Called EHS, talked to Dan Manchaka about contamination spill yesterday
 - d-imenchaca@tamu.edu
 - 979-676-0590
- ☑ EHS came by ~3:20pm to evaluate the contamination in the lab
 - Asked about the incident reported
 - Took pictures of glovebox and room
 - Swiped and surveyed

3 Details from research meeting

- Note that Dr. Chirayath needs a VGA to HDMI converter
- Discussed research results
 - Want the third experiment to be completed
- Discussed contaminaiton

– Specific Activity of $^{239}\text{Pu: }0.063~\frac{Ci}{g},$ largest amount of Pu released: 5 μg

$$0.063 \frac{Ci}{g} \cdot \frac{10^{-6}g}{\mu g} \cdot \frac{3.7 \times 10^{10}Bq}{Ci} = 2331 \frac{Bq}{\mu g}$$
$$2331 \cdot 5\mu g = 11655Bq$$

— Specific Activity of $^{238}\text{U}\text{: }12{,}445~\frac{Bq}{g},$ largest amount of U released: 0.000258 g

$$0.000258 \ g \cdot 12445 \frac{Bq}{g} = 3.21Bq$$

- Annual intake limits $\sim 300~\mathrm{Bq}$
- Say 40% was released to air: 4663 Bq
- Room size is about 72 cubic meters = 72000 liters
- 0.065 Bq/liter
- Human breathes 20 times per minute with 6 liter capacity
- 2 liters per second, 7200 liters per hour

$$0.065 \frac{Bq}{liter} \cdot 7200 \frac{liters}{Hr} = 468 \frac{Bq}{Hr}$$

- Things to discuss with Dan:
 - 1. Ask Dan if a spill procedure should exist for antichamber
 - 2. Remind Dan biggest concern is evaporation
 - 3. Should we get Masks

Thursday, 27 October 2016 9:30 am

- ✓ Update laboratory notebook
- \square Determine calculation for alpha samples
- ☐ Outline project for UQ
- ✓ Meet with Dan Menchaka about rad stuff
 - Called him on the phone
 - He said that swipes came back clean
 - That I could continue to decontaminate in the glovebox
- ✓ Installed uncertainties on windows computer
 - Go to start menu
 - cmd, run in administrator mode
 - type_path_to_pip install package
- ✓ Automated copy paste from Gamma_Template to excel sheet

1 Cycle experiment, replicate of 3

- \mathbf{Z} Finish counting 6 AqII

Friday, 28 October 2016

1 Contamination spill 10/25/16

- ✓ Clean contamination in glovebox
 - Swipe L Shoe clean
 - Swipe R Shoe clean
 - Swipe Top clean
 - Swipe Left Right Mid plane clean
 - Swipe around the top back portion clean
 - Swipe Back bottom clean

2 Cycle experiment, replicate of 3

- ☑ Checked math with John Burns
 - The math was correct, but we noticed that Series 6 had larger D-values across the board
 - If we assume a 10 μ l contamination of aqueous in the organic (a very small amount), the D-values line up a lot better
 - Eu-155 0.07 to 0.049 **☑**
 - Eu-155 0.09 to 0.073 **☒**
 - Eu-154 0.095 to 0.073 **☒**
 - Ce-144 0.045 to 0.022 **☑**
 - Rh 0.067 to 0.045 **☑**
 - Cs-137 0.024 to 0.001

Monday, 31 October 2016

1 Cycle experiment, replicate of 3

- ✓ Start Efficiency Count with Eu-152 Liquid source
- ✓ Stop Efficiency count once contamination was found need to clean HPGe

2 Contamination spill 10/25/16

- ☑ Luis Gonzolas and Daniel Menchaca both came by around 10:00 am to take swipes around the antechamber
 - They said they would get results after lunch
- ✓ Write up small report about contamination leak and give to Latha, in subdirectory "Indicent"
 - \bullet Assumed 90% of the 7 series in the antechamber, and the other 10% is in the original 7 vial that wasn't spilled

3 Minor Contamination of HPGe, found Monday 10/31/2016

- Clean HPGe, reduce background contamination
 - Clean all bricks
 - Count with bricks in different configurations
 - Found that source is coming from radiation storage closet
- Ask Troy if he moved sources around in closet, or if anyone did
 - He did say that someone moved stuff around
 - Shielded our source (probably strongest source around)
- ✓ Recount background, still high on Cs-137 source...
- ✓ Ask Marianno for doubloon reward...and if he aquired any sources recently, he said he did, he got 1.3 or so mCi of ¹³⁷Cs...that would explain it, I asked which day he got the source, to know when to subtract out the background from my samples...he said he would check

Dig around the roots Grace and Truth Next season will come

Tuesday, 1 November 2016

1 Contamination spill 10/25/16

- ☑ Dr. Latha Vasudevan contacted with questions, responded as well as I could
 - She said no more experiments until waste could be picked up
 - She said that vials should be in its own box
- ☑ Contacted EHS about Waste pickup, but need the PI's username and password
 - Sorry Dr. Folden, but I need to bother you about this
 - 1. Start at EHS Website
 - Safety Tab \rightarrow Radiological Safety
 - Request Waste Pickup (link)
 - Link for request at bottom of page
 - 2. Activities should be corrected to the date the smaple was added to the license, assume the date to be May 5th, 2014
 - 3. License number is 933
 - 4. Last time 0.00005 mCi removed, 0.657392 remains

2 Minor Contamination of HPGe, found Monday 10/31/2016

- ☑ Got Dr. Mariannos source list, last time he got ¹³⁷Cs, was in September, not during the time of our experiment he did say that sources were moved around two weeks ago on Thursday
- ☑ Calculation for MDA Modify pages 96-98 from Knoll to do in terms of CPS, not total counts
 - Also looked at Ludlums calculation Ludlum
 - Created a Excel Sheet for example calculations with equations
- ✓ Marianno said that he shielded the ¹³⁷Cs
- ✓ Started a new background count
 - It does look like he shielded ¹³⁷Cs
- ✓ Clean all outside vials

Wednesday, 2 November 2016

1 Cycle experiment, replicate of 3

- ✓ Finish background count
- ☑ Start Efficiency Count with Eu-152 Liquid source, again (on Monday we found the ¹³⁷Cs higher background)
- ☑ Background corrections for all calculations
 - Added Background Row to Gamma_Template, call it now Gamma_Template_BK, this will subtract background
 - Could automate subtraction, need to add this row based on background of background
- \mathbf{Z} Assuming 10μ l contamination what are D-values
- ☑ Checked calculation on why the error for D-values from Aqueous are so bad, mostly due to how its calculated. Calculated a different way, gave same answer, but slightly larger error, I guess I'll have to abandon that type of calculation.
- \square Make Easy to read power point
- ✓ Automate Decay corrections

2 Details from research meeting

- \bullet Showed results, at first Chirayath, thought that $^{137}\mathrm{Cs}$ was not behaving the same, but showed it was
- Said we need to do the experiment three times again, only the extraction

Thursday, 3 November 2016

1 Cycle experiment, replicate of 3

- - The Weighted Mean

$$\hat{\mu} = \frac{\sum x_i / \sigma_i^2}{\sum 1 / \sigma_i^2}$$

$$\sigma^2(\hat{\mu}) = \frac{1}{\Sigma 1/\sigma_i^2}$$

 \square Automate background calculation and decay corrections

2 Contamination spill 10/25/16

- ✓ Talked with Evgeny Tereshatov: ETereshatov@tamu.edu
 - Said 52.50 \pm 0.5 μ Ci decay corrected to 5 May, 2014 ¹⁴⁴Ce is to be disposed
 - RSO 0079436
 - Need Waste Disposal Report Form
 - \bullet Made estimates on $^{137}\mathrm{Cs},\,^{134}\mathrm{Cs}$
 - Accidentally added ⁹⁰Sr, it should have been ¹²⁵Sb
- ☑ Called EHS three times, left message once no response

Friday, 4 November 2016

1 Cycle experiment, replicate of 3

- \mathbf{Z} Dr. Burns suggested to not use Series 7 in the calculations did yesterday, I removed them from the calculations, changed the final result by 0.2 μ l. (10.5 to 10.7)
- ☐ He also suggested to do the correction calculation at an earlier stage, like in the CPS arena, which would take a lot more work honestly I don't think it will change things much, probably the same about as above
- ✓ Automate background correction
 - Will do background correction based on most recent background
 - Should probably change to search for a background date
 - Okay now changed to search for a specific background date
- ✓ Automate Decay corrections

2 Contamination spill 10/25/16

- ✓ Called EHS, no response, found old waste dissposal sheet, filled it in
- ✓ Called Innocent, he said he would come, please come!
- \mathbf{Z} EHS came! Thank you Innocent, he picked up the waste, took the sheet, and gave us new waste bags

3 Cycle experiment, round 2, replicate of 3

- 🗹 Aaron Kruger let me into the Radiation source closet (so I can get more sample)
 - Grabed our source, stored in the back of lab with shielding
- ☑ Complete ¹⁵²Eu count
- ✓ Start background (make sure things are okay)

Monday, 7 November 2016

1 Cycle experiment, round 2, replicate of 3

- ✓ Finish background count
- \square Practice transfer with 300 μ l.
 - A little frustrating
 - Take a lunch break for headache, maybe second practice will go better
 - Settled on 400 μ l instead of 500 μ l or 300 μ l (happy medium)
- ☑ Create and label vials [8], [9], [10], and [Buddy], to hold stock solution. Did not leech them, hopefully barium contamination wont be a huge deal, we will assume all the data for Cs can be gathered from ¹³³Cs. also, still using smaller vials, but will make sure to have double containment for transfer into glovebox
- \square Create \boxed{Buddy} with 0.5 0.4 (removed 0.1) ml of 4 M HNO₃ solution
- ${\bf \not\!\! C}$ Put $\overline{|Buddy|}$ inside a 15 ml vial, parafilm wrap
- **✓** _

2.048+/-0.026 ml of 1.12+/-0.08 M HNO₃ solution $\longrightarrow Stock\ Add$ (glass container)

- Transfer Stock Add, [8], [9], [10], [Buddy], and [closet] to glove box, (with additional 15 ml vials for containers that will need them)
- **✓** -

Combine 0.500+/-0.005 ml of 15.43+/-0.06 M HNO₃ solution
$$\boxed{closet}$$
 + 2.048+/-0.026 ml of 1.12+/-0.08 M HNO₃ solution $\boxed{Stock\ Add}$ = 2.500+/-0.025 ml of 4.00+/-0.05 M HNO₃ solution. $\boxed{\rightarrow\ Stock\ Add}$

_ -

Combine 2.500+/ 0.025 ml of 4.00+/ 0.05 M HNO $_3$ solution. Stock Add + 0.700+/ 0.028 ml of 4.00+/ 0.05 M HNO $_3$ solution Stock = 3.2+/-0.038 ml of 4.00+/-0.05 M HNO $_3$ solution. \rightarrow Stock

- A problem...I am not sure how this happened, and I kind of don't want to bring it up, but I was able to get only, 400 μ l out of \boxed{Stock} , I would expect to get 690 μ l out of \boxed{Stock} ..where did 290 μ l go? Did it evaporate? Do we need to parafilm wrap it?
- As a precaution, I will parafilm wrap it
- \blacksquare Transfer 400 μ l Stock to Stock Add
 - Also switched caps (because aluminum foil cap was removed on Stock and I liked having it off)
- ✓ Transfer *closet* out of glovebox
- \checkmark Transfer 0.4 ml of $\boxed{Stock \ Add}$ to $\boxed{8}$
- \blacksquare Transfer 0.4 ml of $\boxed{Stock \ Add}$ to $\boxed{9}$
- ✓ Transfer 0.4 ml of Stock Add to 10
- ✓ Add scoop of sodium nitrite to 9
- ✓ Add scoop of sodium nitrite to 10
- $\ensuremath{ \mathbb{Z} }$ Put $\ensuremath{ 8}$, $\ensuremath{ 9}$, and $\ensuremath{ 10}$ into 15 ml centrifuge tubes
- ✓ Centrifuged [8], [9] and [10] to push all solution to bottom of vials
- - Retake background and efficiency count
- \square Note when ¹³⁷Cs will be floating around lab
 - T, Th 1-4 pm, and Wed 2-5, this week and next week
 - Do not count during this time
- ☑ Background Count

Monday, 7 November 2016

√	Eff Count
	Practice extraction with 400 μ l while doing counts to night
√	Count 8
1	Count 9
	Count 10
	• Alarm didn't wake me updidn't count 10

Tuesday, 8 November 2016

1 Cycle experiment, round 2, replicate of 3

- ☑ Count 10
- ✓ Label vials 8 mix, 9 mix, 10 mix (smaller 1 ml tubes from John Burns, have conical bottoms, makes more minute separations easier)
- Transfer [8], [9], and [10] into glovebox. With: [8] [aq], [8] [aq], [8] [aq], [8] [aq], [9] [aq], [9] [aq], [9] [aq], [10] [aq], [10] [aq], [10] [aq], [10] [aq], [10] [aq], [10] [aq], [
- \checkmark Add 400 μ l of \boxed{TBP} to $\boxed{8}$, $\boxed{9}$, and $\boxed{10}$ each
- ✓ Vortex mix 8 for 15 minutes on pulse mode
- ✓ Vortex mix 9 for 15 minutes on pulse mode
- ✓ Vortex mix 10 for 15 minutes on pulse mode
 - Switched to push caps for each of the above
- \mathbf{Z} Centrifuge [8], [9], and [10] with [Buddy] on 3300 rpm, for 5 minutes
- \square During the vortex mixing and the centrifuge practice the transfer in the fumehood
 - Was able to get about 395 ml of aqueous phase and 365 ml of organic phase
- Pipette with disposable pipette the aqueous phase first, then the organic (for all three vials), as much as so that there is no mixing. Then transferred the boundary to a smaller vial, centrifuged, and separated further. Counting solutions were also prepared of 250 μ l of each of the solutions **Should have centrifuged final solutions before this**. A picture will be provided for the whole process for $\boxed{8}$ on the following page, below are specific notes about what occured during the experiment.

Tuesday, 8 November 2016

- 10 had to be centrifuged again with Buddy (shock the phases too much so they mixed again accidentally pipetted organic phase during aqueous phase first separation)
- 9 mix, 10 mix had to be recentrifuged (accidentally dropped these two small(!) vials (no place to put them)
- 8 mix Lost a drop while making 250 μ l Aq sample
- 9 mix Lost a drop while making 250 μ l Aq sample
- 10 mix Lost a drop while making 250 μ l Aq sample
- \square Measure volumes of everything

- ✓ Clean stuff in glovebox

- ✓ Create graphic for experiment

Tuesday, 8 November 2016



Extraction three times round 2 experimental setup

Wednesday, 9 November 2016

1 Cycle experiment, round 2, replicate of 3

- - Gave decent results for everything but ¹³⁷Cs

- ✓ Analyze results from experiment, display in a single excel sheet
 - Note GENIE corrects for dead time, but if you had to do it by hand, here is the equation for small corrections

$$CPS_f = \frac{CPS_i}{1 - \frac{DT}{100}}$$

2 Details from research meeting

- Perfect ¹³⁷Cs
- $\bullet~{\rm Fix}~^{154}{\rm Eu}$
- MARLAP, Stat teaching, look up MDA
- Submit Degree plan, put a policy course on there
- Subtracting BK is why I go negative sometimes, another reason for negative values in the D-value is because sometimes I take a difference
- Covariance data is MT133

Thursday, 10 November 2016

1 Cycle experiment, round 2, replicate of 3

- Finish count 9 or C
- \square Start count $\boxed{8 \text{ or } C}$ on face of detector ($\sim 4 \text{ pm}$)
- ☑ Check if a geometric constant correction factor can be applied for the second geometry
 - It can kind of be applied...but not really
- ☑ Looking at CPS for ¹³⁴Cs between aqueous before extraction, and after (need to include volumes in calculation because each solution had different volumes)
 - Did calculation for MDA, visually showed why we cant use the information...unless we count for a longer time

2 Things to do for school

- ☐ Alpha analysis
- \square Respond to McClarren email did this weekend
- \square Review McClarrens notes email did this weekend
- \square Learn how to use ORIGEN did this weekend

Friday, 11 November 2016

1 Cycle experiment, round 2, replicate of 3

☑ Start Efficiency count on face of detector

2 Things to do for school

 \square Find variances

✓ Learn how to use ORIGEN, and run it

 \Box Come up with chaos polynomial plan

Do a write up for McClarren...so it looks like I am doing work for his class

Monday, 14 November 2016

1 Cycle experiment, round 2, replicate of 3

- Finish Efficiency count on face of detector
- ✓ Analyze results...something is very fishy
 - \bullet The $^{137}\mathrm{Cs}$ results look very small $10^{\text{-}5}.$ Which we were hoping they would be around 0.01
 - The other results had higher D-values across the board maybe due to geometric differences, maybe not
- - To check and see if geometry is the issue (although these high dead times would probably give incorrect results as Dr. Burns pointed out)
- - Also noticed that Series 9 is a little funky...its always higher in D calculations by a large portion (except for Cs, where its lower)
 - Frustrating!
 - Dr. Kitcher brought up the issue that could be correcting to the wrong value (in series 6) should find literature values
 - Dr. Burns brought up that the fact that I didn't centrifuge the samples during the last step could be the issue
 - Web of Knowledge, Web of Science, Periodic Table.com
- - Transfered above vials into glovebox after parafilm wrapping
 - Took all solution out of above vials, and put into original containers (labeled the same without the C $\boxed{8 \text{ or}}$ as opposed to $\boxed{8 \text{ or } C}$)
 - Centrifuged both C and non-C containers for 5 minutes on highest setting (33)

- Repipetted out 250 μ l out of non-C containers into C containers
- Put C containers into 15 ml centrifuge tube
- Transfer out of glovebox and clean

☑ Dr. Burns found a reference with some useful data, Link, table from reference below

Quick calculation for molarity of uranium in samples

$$\frac{0.0129 \text{ g DUO}_2 \cdot 0.88 \cdot \frac{1}{238}}{0.005 \text{ Liters}} = 0.009539 M$$

$$0.009539 \text{ M} \cdot \frac{0.5 \text{ } ml}{2.5 \text{ } ml}$$

$$= 0.001908 \text{ M U}$$

Quick calculation for saturation of uranyl nitrate in water at 20 °C.

$$\frac{122 \text{ g}}{\text{g}} = \frac{122 \text{ g}}{\text{g}} \cdot \frac{1000 \text{ } ml}{L} \cdot \frac{mol}{394.04 \text{ } g} \approx 3.09 \text{M}$$

Values from Paper, 1.4 M U (much higher than ours 2 mM), and 3 M HNO₃ (ours is at 4 M)

Element	D-Value
Ru	0.04
Rh	0.01
Pd	0.09
Nd	0.04
Ce	0.02
Sr	0.00
Sm	0.07
Cs	0.01

The below figure shows that as the concentration HNO_3 increases from 3 to 4, we shouldn't expect a huge difference between reference and our values. Literature values should have some difference between 0% uranium saturation to 45% saturation (reference)



D value plots from Reactor handbook

- Ru, and Ce match from our experimental results from the first experiment, and Cs is around 0.01...which is what we are looking for, Sr there is no number (except that its small, which is in line with our first experiments). I also want to point out, no error bars, Dr. Folden would be not be happy, these numbers don't mean anything
- \checkmark Looking at geometric differences between calibration source at 0 cm and 26 cm. and also between $\boxed{10 \ or}$ at 0 cm and 26 cm.
 - Noticed there is a trend, might be able to use
 - Also noticed that I counted my ¹⁵²Eu source at 26 cm for a short time (1.9 live time hours)... will start count for that in the morning and count while doing the experiment, maybe that will fix some problems. The reason for this short count time, is I feel lots of pressure to finish

Tuesday, 15 November 2016

1 Cycle experiment, round 2, replicate of 3

- Start efficency count at 26 cm around 5:50 am
- - Counts for Ce, look better, Eu look better, Ru look worse, Cs look better (but still one order of magnitude off
 - Looking into the count rates, some peaks change by alot between the first and second count of 10 or C and the second...WHY!?
 - Maybe because some aqueous was in the original sample 10 or, and because it had some time to dissolve into the solution, the activities for the lower D materials increased

2 Cycle experiment, round 2, replicate of 3, ALPHA preparation for extraction 1 (also Mass Spec preparation)

3 Cycle experiment, round 2, replicate of 3

- ✓ Finish Eff count
 - Rework calculations with new eff...didn't help much
- ✓ Start 9 or count

Tuesday, 15 November 2016

- ✓ Spend all night making spreadsheet to calculate how much volume would be optimal for contamination in each series
 - It made things kind of work better, but not a whole lot better
 - Reason why I haven't averaged numbers yet...was taking a 26 counting efficency, counted most of the day
 - \bullet Also determined geometric differences between calculating activity at 0 cm as opposed to 26 cm there wasn't much of a difference
 - Also, need to complete recounts for $\boxed{8 \text{ or } C}$ and $\boxed{9 \text{ or } C}$

Wednesday, 16 November 2016

- ✓ Transfer in the glovebox a blue 2.5 ml vial (also hold smaller conical vials) holder sorry Mary, it makes things much easier to have something to hold your vials
- ✓ Transfer smaller pipette tips into glovebox

1 Cycle experiment, round 2, replicate of 3

- ✓ Finish count 9 or

2 Cycle experiment, round 2, replicate of 3, ALPHA preparation for extraction 1 (also Mass Spec preparation)

✓ Make alpha sample of stock, make 3 (Pipette Errors - assume 20 μ l error 1%, 10 μ l error 1.2%, 390 μ l error 2%, 890 μ l error 1%)

10+/-0.12
$$\mu$$
l of Stock Add (4 M HNO₃) [smaller pipette] + 990+/-9.9 μ l of DI water (leftover in glovebox) = 1+/-9.9 ml of \sim 0 M HNO₃ $\boxed{8,9,10~Dilution}$

$$20+/-0.2~\mu l$$
 of $\boxed{8,9,19~aq~Dilution}$ dropped onto $\boxed{8~Chip}$ $20+/-0.2~\mu l$ of $\boxed{8,9,19~aq~Dilution}$ dropped onto $\boxed{9~Chip}$ $20+/-0.2~\mu l$ of $\boxed{8,9,19~aq~Dilution}$ dropped onto $\boxed{10~Chip}$

Wednesday, 16 November 2016

10+/-0.12
$$\mu$$
l of $\boxed{8~aq}$ (4 M HNO₃) [smaller pipette] + 390+/-7.8 μ l of DI water (leftover in glovebox) = 0.4+/-0.0078 ml of \sim 0 M HNO₃ $\boxed{8~aq~Dilution}$

• 8 aq transfer contaminated gloves (had the blue push cap) and the vial accidentally fell

$$20+/-0.2~\mu l$$
 of $\boxed{8~aq~Dilution}$ dropped onto $\boxed{8~aq~Chip}$

✓ - 9 aq

• $\boxed{9~aq}$ and $\boxed{10~aq}$ centrifuged, so no contamination on glovebox gloves like above

$$10+/-0.12~\mu l$$
 of $\boxed{9~aq}$ (4 M HNO₃) [smaller pipette] + $390+/-7.8~\mu l$ of DI water (leftover in glovebox) = $0.4+/-0.0078~m l$ of $\sim 0~M~HNO_3$ $\boxed{9~aq~Dilution}$

$$20+/-0.2 \mu l$$
 of $9 aq Dilution$ dropped onto $9 aq Chip$

☑ - 10 aq

10+/-0.12
$$\mu$$
l of $\boxed{10~aq}$ (4 M HNO₃) [smaller pipette] + 390+/-7.8 μ l of DI water (leftover in glovebox) = 0.4+/-0.0078 ml of \sim 0 M HNO₃ $\boxed{10~aq~Dilution}$

$$20+/-0.2~\mu l$$
 of $\boxed{10~aq~Dilution}$ dropped onto $\boxed{10~aq~Chip}$

10+/-0.12
$$\mu$$
l of $\boxed{8\ or}$ (30% TBP) [smaller pipette] + 890+/-8.9 μ l of 30% TBP (leftover in glovebox) = 0.9+/-0.0089 ml of 30% TBP $\boxed{8\ or\ Dilution}$

$$20+/-0.2~\mu l$$
 of $\boxed{8~or~Dilution}$ dropped onto $\boxed{8~or~Chip}$

• Spilled some organic on inner ring?? of 8 or Chip, question because hard to see in glovebox

✓ -
$$\boxed{9\ or}$$

$$10+/-0.12\ \mu\text{l of }\boxed{9\ or}\ (30\%\ \text{TBP})\ [\text{smaller pipette}]$$
+
$$890+/-8.9\ \mu\text{l of }30\%\ \text{TBP (leftover in glovebox)}$$
=
$$0.9+/-0.0089\ \text{ml of }30\%\ \text{TBP }\boxed{9\ or\ Dilution}$$

$$\mathbf{10}+/-0.12~\mu l$$
 of $\boxed{9~or~Dilution}$ dropped onto $\boxed{9~or~Chip}$

• Changed volume on chip because 8 or Chip potentially spilled over the inner ring

✓ -
$$\boxed{10~or}$$

 $10+/\text{-}0.12~\mu\text{l of }\boxed{10~or}$ (30% TBP) [smaller pipette] + 890+/-8.9 $\mu\text{l of }30\%$ TBP (leftover in glovebox) = 0.9+/-0.0089 ml of 30% TBP $\boxed{10~or~Dilution}$

• Changed volume on chip because 8 or Chip potentially spilled over the inner ring

 $10+/-0.12 \mu l$ of 10 or Dilution dropped onto 10 or Chip

- ✓ Note: Centrifuged all dilution vials before making alpha samples, which means that first all dilutions were made, then all alpha samples were made
- ☑ The above 7 alpha samples take up space in the glovebox, and I didn't want to disturb the samples (moving them screws them up) so I let them dry overnight

3 Process Experiment (continuation from cycle experiment)

 ${f {\it Z}}$ Combine all aqueous phases together (done with disposable pipetets)

$$\mathbf{Z} \ 8 \ aq \ C + 8 \ mix \rightarrow 8 \ aq \ (take all of first and add to second)$$

$$\boxed{10 \ aq \ C} + \boxed{10 \ mix} \rightarrow \boxed{10 \ aq}$$

4 Details from research meeting

- Just present D-Values at research meeting
- Things didn't add up so well
- Dr. Chirayath didn't like my ¹³⁷Cs values, looked at the first experiment, the one where I messed up, and liked the 110 value, now I am getting 10⁻⁵...why?
- \bullet Dr. Burns suggested to increase the volume of the extraction phase (organic) to pin down the $^{137}\mathrm{Cs}$ values
- Dr. Folden also said that we should average the percent extraction values, not the D-values, because D values vary widly at the ends (shown in next figure)

$$Fraction\ Extracted = \frac{Mass\ Organic}{Mass\ Initial}$$

- Dr. Chirayath said to continue process
- Jeremy had interesting results, the flux spectra turned from kind of fast to thermal, Gd burned out
- Robert Zedric also noted that a higher dead time could be used, and that our detector is between a Nonparalyzable and paralyzable model, and that we could try to work through the math on that, Knoll page 122



Percent extraction versus D value on log scale

Thursday, 17 November 2016

1 Cycle experiment, round 2, replicate of 3, ALPHA preparation for extraction 1 (also Mass Spec preparation)

Note all alpha counts were done on the 9mm height setting on the pips detector.

- ✓ Start Count 10 or Chip (10:52 am)
- ✓ End Count 10 or Chip Run time 7.54 hrs

2 Experiment to double check ¹³⁷Cs using combined aqueous series 5 and 6, 56

In order to capture the D-value for ¹³⁷Cs, an experiment was proposed. Our problem with measuring ¹³⁷Cs is that its D-value and activity are so low that we aren't getting good statistics for its answer, and the answer we are getting is not the answer we want, we are getting something around 10⁻⁵, and the answer is more probably around 0.01.

It was proposed to take an old series (series 5 or 6), and perform an extraction with a larger volume of organic, so that more ¹³⁷Cs could be extracted, and therefore better statistics on all the calculations. Some notes are copied down from hand calculations for the experiment.

- $\boxed{5~aq}$ has 461 μ l, 4.47 μ Ci, $\sim 3.6\%$ dead time
- 6 aq has 469 μ l, 4.40 μ Ci, $\sim 3.6\%$ dead time, this vial is also a little milky, meaning there is a small amount of organic in there
- Both above vials should were in fumehood
- Some evaporation happened in Stock, I know this because the activity density changed from Stock and Stock add.
- If we take 800 μ l total (after mixing 5 aq and 6 aq), then we could expect \sim 8.87 μ l (about 200 cps), of ¹³⁷Cs with \sim 6% dead time

• If we want 3 cps in the final organic (about an hour of count time) and if I assume the D-value is 0.01 (which Dr. Chirayath insists), $(3/200 \sim 1.5\%)$ of the counts)

$$\% = \frac{1}{1 + \frac{V_a}{V_o} \frac{1}{D}}$$
$$= \frac{1}{1 + \frac{1}{2} \frac{1}{0.01}} = 0.019$$

This means if we double the volume of the organic, then we should get a decent count rate so as to count ¹³⁷Cs and get good statistics with an hour count. This is IF the D-value is 0.01, as Dr. Chirayath insists.

- Dr. Burns came by and said, instead of 2x the organic volume, should do 10x, to make sure we get all the counts!
- Okay! Sounds good! We will for sure get the right answer now! We also rederived the D-value equation

With conservation of mass, and using values from the two phases,

% Extracted =
$$\frac{\left[\frac{CPS}{V_m}\right]_o \cdot V_{co}}{\left[\frac{CPS}{V_m}\right]_o \cdot V_{co} + \left[\frac{CPS}{V_m}\right]_a \cdot V_{ca}}$$

$$\frac{1}{\% \text{ Extracted}} = \frac{\left[\frac{CPS}{V_m}\right]_o \cdot V_{co} + \left[\frac{CPS}{V_m}\right]_a \cdot V_{ca}}{\left[\frac{CPS}{V_m}\right]_o \cdot V_{co}}$$

$$=1+\frac{[\frac{CPS}{V_m}]_a\cdot V_{ca}}{[\frac{CPS}{V_m}]_o\cdot V_{co}}$$

$$=1 + \frac{1}{D} \cdot \frac{V_{ca}}{V_{co}}$$

$$\frac{1}{\frac{V_{co}}{V_{co}} \left(\frac{1}{\% \text{ Extracted}} - 1 \right)} = D$$

Where V_m is the measured volume for the count, V_{co} is the volume of the organic contact and V_{ao} is the volume of the aqueous contact.

- Take 800 μ l out of 5 aq and transfer into a 15 ml vial labeded 56 (for some reason it was really difficult to get a precise volume had to do many times)
- ✓ Start count 56 at 26 cm

3 Cycle experiment, round 2, replicate of 3

- ✓ Analyzed last two organics, put into excel sheet
 - All samples of organic, after mixing organic parts together, redrawing 250 μ l and recounting, increased in activity. This could support the conclusion that some aqueous passed to the main organic, and when the 250 μ l was first drawn, was on the bottom of the vial. When the 250 μ l was second drawn, it had time to dissolve into the TBP, because HNO₃ is slightly soluble in TBP (Nuclear Chemical Engeineering pg 160)

4 Process Experiment (continuation from cycle experiment)

Volumes for combined aqueous phases

Series	Aqueous (8,9, or 10)
8	397 + / -7.94
9	$386\ 389 + / -7.78$ (after centrifuge)
10	395 +/- 7.9

Second Contact...

- - Will reuse 8 mix, 9 mix, 10 mix (smaller 1 ml tubes from John Burns, have conical bottoms, makes more minute separations easier)
- ✓ Transfer: $8 \ aqII$, $8 \ aqII \ C$, $8 \ orII$, $8 \ orII \ C$ $9 \ aqII$, $9 \ aqII \ C$, $9 \ orII$, $9 \ orII$, $9 \ orII$, $10 \ aqII \ C$, $10 \ orII$, $10 \ orII \ C$. (3 clear push caps, and 6 blue push caps 6 red push caps). Also with 6 15 ml centrifuge tubes

Thursday, 17 November 2016

√	Vortex mix $8 aq$ for 15 minutes on pulse mode				
√	Centrifuge 8 aq with Buddy at 3,300 rpm for 10 minutes • Decided after this to wait, and centrifuge them all together				
√	Vortex mix $9 aq$ for 15 minutes on pulse mode				
✓	Vortex mix $\boxed{10~aq}$ for 15 minutes on pulse mode				
√	Centrifuge $\boxed{8\ aq}$, $\boxed{9\ aq}$, and $\boxed{10\ aq}$ with \boxed{Buddy} on 3300 rpm, for 5 minutes				
	During the vortex mixing and the centrifuge practice the transfer in the fumehood Prayed instead				
	Pipette with disposable pipette the organic phase first, then the aqueous (for all three vials), as much as so that there is no mixing. Then transferred the boundary to a smaller vial, let sit. Prepare counting solutions of 250 μ l of each of the solutions A picture will be provided for the whole process for $8 \ aq$ on the following page, below are specific notes about what occured during the experiment.				
	• $8 \ aq$ was 248 μ l pipetted to $8 \ aqII \ C$ instead of 250 μ l?				
	Measure volumes of everything				
√	Transfer out $\boxed{8\ or II\ C}$, $\boxed{8\ aq II\ C}$, $\boxed{9\ or II\ C}$, $\boxed{9\ aq II\ C}$, $\boxed{10\ or II\ C}$, $\boxed{10\ aq II\ C}$, in 15 ml centrifuge tubes				
√	Radiac wash the above tubes, and store in fumehood behind lead - wait to count				
√	Clean stuff in glovebox				
V	Start count 9 or II C at 0 cm 4:06 pm				

Thursday, 17 November 2016



Extraction three times round 2 extraction 2

Friday, 18 November 2016

1 Cycle experiment, round 2, replicate of 3, ALPHA preparation for extraction 1 (also Mass Spec preparation)

Note all alpha counts were done on the 9mm height setting on the pips detector.

- ☑ End Count 10 aq Chip Run time 14.4 hrs
- ✓ Start Count 9 or Chip (9:02 am)
- End Count 9 or Chip Run time 6.08 hrs
- ✓ Start count 9 aq Chip (3:11 pm)

2 Process Experiment (continuation from cycle experiment)

3 Experiment to double check ¹³⁷Cs using combined aqueous series 5 and 6, 56

Talked about volume changes with Kevin, using 50 ml tubes for the whole experiment

- Transfer 56 into glovebox with labeled 50 ml tubes $56 \ Big$, $56 \ Big \ Aq$, and $56 \ Big \ or$
- ${\bf Z}$ Take the 800 μ l out of ${\bf 56}$, and transfer to ${\bf 56}$ Big (had to do middle step of transfering everything to ${\bf 5}$ aq)

 ${\bf Z}$ Start count $66 \ Big$ (11:17 am) to around 1:04 (started count $60 \ orline{10}$

Just prior to stopping the above count, Dr. Burns suggested keeping all 800 μ l of the aqueous in 56 instead of 56 Big and just but all organic into a 50 ml tube. So now...

- \square Transfer $56 \ Big$ into glovebox (wrapped in a ziplock bag so that less evaporation)
- \square Transfer 800 μ l out of 56 Big into 56
- \checkmark Add 8.0 μ l of TBP to $\boxed{56}$
- ✓ Shake 56 on vortex mixer for 15 minutes
- Convert a 2.5 ml vial holder (a 15 ml tube) to a buddy, by adding 800 μ l of DI water and 8.0 ml of TBP to it...scratch out label. $56 \ Buddy$

- ☑ Clean up work area in glovebox
- \square Transfer interface of $\boxed{56}$ to $\boxed{56 \ mix}$, seal and let sit for the time being
- \checkmark Transfer out of glovebox $\boxed{56~aq}$ and $\boxed{56~or}$. Clean with radiac wipes

$$\frac{200~{\rm cps_{aq}}}{800~\mu l} \cdot 8,000~\mu l \cdot 0.01 = 20~{\rm cps_{or}}$$

Sadly, first glance gives around $0.1~\mathrm{cps_{or}}$, I have failed again. Sorry Dr. Chirayath. I am feeling fairly defeated, I just want to go home.

Sunday, 20 November 2016

1 Cycle experiment, round 2, replicate of 3, ALPHA preparation for extraction 1 (also Mass Spec preparation)

Note all alpha counts were done on the 9mm height setting on the pips detector.

- ${\bf \not\!\! C}$ End Count $\boxed{9~aq~Chip}$ Run time 18.5 hrs
- **☑** Start Count 10 *Chip* (1:34 pm)

2 Experiment to double check ¹³⁷Cs using combined aqueous series 5 and 6, 56

- ✓ End count 56 or

Monday, 21 November 2016

- Dr. Mariannos experiment today started around 3:00 pm

1 Cycle experiment, round 2, replicate of 3, ALPHA preparation for extraction 1 (also Mass Spec preparation)

Note all alpha counts were done on the 9mm height setting on the pips detector.

End Count 10 Chip Run time 18.5 hrs

☑ End Count 9 Chip Runtime 5.3 hrs

2 Process Experiment (continuation from cycle experiment)

Reason why there is a "gap" in counting is that there is another experiment going on, and was counting that one.

Tuesday, 22 November 2016

Modify	spreadsheet s	so that	the	three	${\rm errors}$	can	be	minimized	l

☐ Find references for D-values

1 Cycle experiment, round 2, replicate of 3, ALPHA preparation for extraction 1 (also Mass Spec preparation)

Note all alpha counts were done on the 9mm height setting on the pips detector.

$$\mathbf{Z}$$
 End Count $abla aq Chip$ Run time 17.1 hrs

2 Process Experiment (continuation from cycle experiment)

• Start Count from other experiment

3 Experiment to double check ¹³⁷Cs using combined aqueous series 5 and 6, 56

- $\hfill\Box$ Start with analysis of first extraction
 - \bullet Things are discouraging... still getting D of $10^{\text{-}5}$ for calculation using organic and calculation using a queous
 - The other elements are within reason

Tuesday, 22 November 2016

- What is different between my experiments and Jarrod's past experiments?
- \bullet Also note, 15 ml tube has about 3% difference from 50 ml tube
- ${\bf Z}$ Create new TBP, 15 ml TBP + 35 ml kerosene $\rightarrow {\bf T}{BP\ Remake}$
- \checkmark Transfer $\boxed{TBP\ Remake}$ and $\boxed{56\ aq}$ into glovebox
- \square Measured volume of $\boxed{56 \ aq}$ to be 700 μ l...why it so low? We had some evaporation?

- \checkmark Transfer $56 \ AqII$ and $56 \ OrII$ out of glovebox, clean with radiac wipes
- \mathbf{Z} Start counting $56 \ OrII$ at 26 cm away from detector
 - \bullet Initially looks like the sample still has $10^{\text{-}5}$ for $^{137}\mathrm{Cs}$

Wednesday, 23 November 2016

1 Experiment to double check ¹³⁷Cs using combined aqueous series 5 and 6, 56

- ☐ Analyze data from 56 experiment

2 Process Experiment (continuation from cycle experiment)

- \mathbf{Z} Start Count $\boxed{10 \ aqII \ C}$
- \square Analyze second extraction data

3 Cycle experiment, round 2, replicate of 3, ALPHA preparation for extraction 1 (also Mass Spec preparation)

Note all alpha counts were done on the 9mm height setting on the pips detector.

- ☐ End Count 8 or Chip
 - Accidentally cleared data...stupid, recounting
- ☐ Analyze Data from alpha spectrum

4 Analysis for Process 1 Mistake (Gamma)

So I keep getting 10⁻⁵ for Cs, but the first experiment I got 110...what is with that. Went back, I realized for that first experiment I forgot to subtract background, which changed the 110 number to 10⁻⁴. Ah that explains it...but that is still an order of magnitude off. What is the deal?

My ratio of numbers is first solution over last solution. I am comparing this number to a D value, which they aren't exactly the same. So I went through the math, assuming a D value of 10^{-5} and found what the ratio of first solution to last solution should be...and

that number is...10⁻⁴. WOAH! Math works, yes! Talked to Dr. Chirayath about this, we looked up a paper and their number was 10⁻⁴. They are reporting a different D-value though, that needs to convert with densities of solutions (luckily enough they report that information). Which should give us the same numbers.

Now the final question, why is the first number I reported so much different from this final number? I think the answer lies in centrifuging...if we assume a small aqueous contaminant, then we should have agreement with our published paper.

Also this will show that there is nothing wrong with my published paper, we reported a **DF** value for a **process**, we described our process very well, and the small contamination was a result of the process.

Also note that HNO_3 is extracted, meaning our acid concentration is changing a little

Break 24-27, November 2016

1 Process Experiment (continuation from cycle experiment)

✓ Stop Count 10 aqII C

2 Cycle experiment, round 2, replicate of 3, ALPHA preparation for extraction 1 (also Mass Spec preparation)

Note all alpha counts were done on the 9mm height setting on the pips detector.

3 Things to do for school

- ☑ Worked on project for NUEN647...still have a long way to go, but hopefully can finish in a week
- 🗹 Also worked some on setting up my new computer at home
- ☑ Worked on encrypting information on linux systems, wrote a program that will manage that some, but still need to put some more time into it
- Also struggled a lot this break with issues of my family, why is my family life so hard, why does my older brother yell at my other brothers and mom, waving a gun around, why is my dad still in prison 7 years after serving the judge appointed sentance of 3 years...why is the place where he is staying so rude to him so now he's lost sight in an eye. God took away Saul's sight for a time, Jesus said to cast your eye from you if it causes you to sin. Why can't I deal with these emotions of self hatered, and why do I want to kill myself? I hate this, I hate this, I want to quit but I'm afraid if I leave that it would break my family once again, and I don't think they would recover from it

Monday 28, November 2016

1 Experiment to double check ¹³⁷Cs using combined aqueous series 5 and 6, 56

- - Had to count the aqueous phase first, then analyzed
 - Results support a D value for Cs to be around 10⁻⁵
 - Some of the D-values for other elements were somewhat different
- Z Review Dr. Chirayath's paper

Summarize Paper

	D-Value (them)	D-Value (us)
Ru	0.0024	0.05
Се	0.0047	0.03
Cs	1.3e-4	3e-5

Comparison

Condition	(them)	(us)	Effect (theirs should be)
Temp	333 K	298	Lower (less 'bonding')
HNO_3	3.9 M	4 M	\mathbf{Same}
U	$0.12~\mathrm{M}$	$0.002 \; \mathrm{M}$	Lower (TBP taken up)

The difference between 1e-4 and 5e-5 is the difference between 99.99% and 99.995%, a small difference.

- - Reconcile document
 - With 2.35 μ l of aqueous in organic, which would be 0.43% of the aqueous phase, All DF values (except 1) that we are measuring by Gamma would be similar (within error) to what we are currently getting for D.

• The exception is Ru, which would be consistent if we were getting a D value of 0.01, instead we are getting a D-value of 0.03-0.07. Which is a difference between 99% and 93-97%.

2 Process Experiment (continuation from cycle experiment)

- ✓ Start background count
- ✓ Analyze second extraction data
 - Used count from 250 μ l and scaled up to the 390s (volume)
 - Noticed some D-values were significantly higher in the second extraction tried VERY hard not to contaminate
 - Ce $0.03 \to 0.12 \ (97\% \ \text{to} \ 89\% \ [\text{in aq}])$
 - Eu $0.06 \rightarrow 0.22 \ (94\% \ \text{to} \ 82\% \ [\text{in aq}])$
 - $\bullet~{\rm Am}~0.04 \rightarrow 0.18~(96\%~{\rm to}~84\%~[{\rm in}~{\rm aq}])$
 - Ru 0.05 to 0.07
 - Cs 3e-5 3e-5
 - Sb 0.002 to 0.003
 - Maybe difference of species in solution cause difference in D-vlaues?
 - A paper I found stated that some D-values fall considerably with addition of uranyl nitrite to system, because TBP is taken up
 - \bullet From 0 M to 0.004 M D of Zr changed from 0.055 0.05
 - We change from 0.002 to 0.000083 M (guess)
 - Also note that the previous paper was published with 0.001 M U
 - Another paper says that Ce is extracted via Ce(NO₃)₃ ·3TBP

Tuesday 29, November 2016

Some of yesterday over lapped to today, also it took me a while to work out the alpha analysis.

1 Cycle experiment, round 2, replicate of 3, ALPHA preparation for extraction 1 (also Mass Spec preparation)

✓ Analyze Data from alpha spectrum

• ²³⁹Pu and ²⁴⁰Pu have over lapping peaks, if we assume that the 93% is 239 and the rest is 240, we get the results shown in Alpha_Results, also using the following equation for a sum peak.

$$g_1 = \frac{CPS \cdot M_1}{\epsilon N_A [\lambda_1 + \lambda_2 (1/N\%_1 - 1)]}$$

- Subtracted grass from aqueous phases
- Results...are way off

Wednesday 30, November 2016

✓ Update notebook

✓ Mess around some with some of the resuls to make them look better

✓ Compile things to present to meeting

• equations used

$$D = \frac{1}{\frac{V_o}{V_a} \left(\frac{1}{0\%} - 1\right)}$$

$$O\% = \frac{1}{\frac{1}{\frac{v_o}{v_a}D} + 1}$$

Err for calculating O% from D value

$$\sigma_{O\%} = \frac{\sigma_D}{(D+1)^2}$$

✓ Talk about the upcoming months

- Concerned about alpha sample not giving reulsts expected, after second extraction want to do alpha samples, but I want to make sure I can get good alpha results for the first extraction, problem is alpha samples take a while to count
- Concerned about changing D-values, second extraction seems to have different D-values for some elements, should I be concerned about this?
- Concerned about not making timeline, willing to work without pay next semester but would like to finish coursework this semester (failed course and current course), would like to take December off

□ Next steps

- Get alpha values working (I'm okayish with chaning D-values
- Make alpha samples for first extraction
- Third extraction

1 Details from research meeting

Not chronological order.

- ✓ Two extractions with larger volume
 - Support low Cs extraction D-value
 - Other D-values similar to what we were getting before
- ✓ Reconcile first experiment of semester
 - Forgot to subtract out background, changed ratio showed to 10⁻⁴
 - Simulate process for first experiment, with assumed D value of 10⁻⁵, the ratio from first to last solution, should be 10⁻⁴ meaning my experiments from this semester are...sort of consistent
- ✓ Reconcile published paper
 - With 2.35 μ l of aqueous in organic, which would be 0.43% of the aqueous phase, All DF values (except 1) that we are measuring by Gamma would be similar (within error) to what we are currently getting for D.
 - The exception is Ru, which would be consistent if we were getting a D value of 0.01, instead we are getting a D-value of 0.03-0.07. Which is a difference between 99% and 93-97%.
- ✓ Prepare alpha samples for first extraction
- ✓ Analyze alpha samples from first extraction
 - Issue, what do I do about these terrible results?
 - Re-make alpha samples? and recount? will take some time
 - No activity balance
 - Really low organic counts
 - Should I wait on third extraction for this?
- ✓ Analyze alpha results from Mess up
- ✓ Finish counts for first extraction
- ✓ Second extraction
- ✓ Count second extraction
- ✓ Analyze second extraction
 - Much different D-values for three elements

If everything worked out the first time, and made sense, things would move a lot quicker, problem is, there is always a problem.

Thursday 1, December 2016

- \square Check on the items from Research meeting
 - ✓ Check for alpha calculation, mass or atom percent
 - I was using mass percent as atom percent for ²³⁹Pu and ²⁴⁰Pu
 - Updated my calculation, and did not change the results much
 - ☑ Email Dr. Folden asking about an IA position for CHEM102, hopefully it will pay for tuition and insurance
 - ✓ Dr. Chirayath said that maybe they remove all FP at once, and increase recovery later, maybe its a one shot thing
 - - ¹⁰⁶Ru decays to the ground state of ¹⁰⁶Rh, which has a 30 second half-life Decay Scheme
 - Did quick silly calculation, yes we are in secular equilibrium

$$N_h = \frac{\lambda_u N_{uo}}{\lambda_h - \lambda_u} \left(e^{-\lambda_u t} - e^{-\lambda_h t} \right) + N_{ho} e^{-\lambda_h t}$$

- ☑ Check on Molarity of TBP, its 2 mols of TBP per 1 M uranium, will this change the extraction?
 - $\rho_{TBP} = 0.9790, \, \rho_{kerosene} = 0.775 0.840$
 - \bullet TBP was made with 15 ml TBP + 35 ml kerosene

15 ml TBP ·
$$\frac{0.9790 \text{ g}}{\text{ml}}$$
 · $\frac{\text{mol}}{266.32 \text{ M TBP}}$ · $\frac{1}{0.05 \text{ L}} = 1.1028 \text{ M U}$

Before we calcualted molarity of uranium in the solution is 0.002 M Uranium, which goes to 0.0001 M uranium.

- ☐ What if nitric acid concentration changes, and we change D-values?
- \square What is the saturation of Urynal nitrate in TBP?
 - Dr. Burns says not to worry about it, we have 500 times more TBP, I tend to agree
- \Box Got paper for Ruthenium and Zr chemistry, they have some interesting equations on page 20/46, should look into, but need to do some stuff in the lab

1 Cycle experiment, round 2, replicate of 3, ALPHA EXTRACTION II (also Mass Spec preparation)



- ${\bf \not\!\! C}$ Also transfer 3 red and 4 blue push caps for smaller vials
- ☑ Discovered (via Kevin) that each alpha sample needs to be diluted by a factor of 10,000 from the original glovebox solution (not my initial)

2 Things to do for school

- □ Dr. McClarrens project for NUEN647
 - But can't because doing research, I feel like my professor is like, please fail your course.

Friday 2, December 2016

1 Cycle experiment, round 2, replicate of 3, ALPHA EXTRACTION II (also Mass Spec preparation)

- ✓ Asked a question while doing the experiment
 - The final volume of all the calculations is correct, and even though there is a large density change for the 10 μ l of concentrated solution, it doesn't make a difference in the final calculation
- ✓ Make dilution of each aqueous
 - \bullet For 1000 0.6% error, 500 1.0% error, yellow top with 10 μl 3% error

$$10+/\text{-}0.3~\mu\text{l}$$
 of $\boxed{8~aqII}$ (4 M HNO_3) [smaller pipette] $+$ 990+/-5.94 μl of DI water (leftover in glovebox)

1.0+/-0.006 ml of
$$\sim$$
 0 M HNO_3 $\c|\Bar{8~aqII~Dilution}$

$$10+/\text{-}0.3~\mu\text{l}$$
 of $\boxed{9~aqII}$ (4 M HNO_3) [smaller pipette] $+$ 990+/-5.9 μl of DI water (leftover in glovebox)

=
$$1.0+/-0.006$$
 ml of ~ 0 M HNO₃ 9 aqII Dilution

10+/-0.3
$$\mu$$
l of 10 $aqII$ (4 M HNO₃) [smaller pipette] + 990+/-7.8 μ l of DI water (leftover in glovebox)

=
$$1.0+/-0.006 \text{ ml of} \sim 0 \text{ M HNO}_3 10 \text{ aqII Dilution}$$

 \square Make dilution of each organic phase

Friday 2, December 2016

2 Experiment to double check ¹³⁷Cs using combined aqueous series 5 and 6, 56

☑ Do a third extraction of 56, measureing volume, and using the original TBP

- Measure $\boxed{56~aqII}$ at 572 μl of aq
 - We keep losing alot of Aq...why?!
 - Is the organic gaining in volume with nitrate going to TBP?
- Add 5.72 ml of TBP (first solution) to $\boxed{56 \ AqII}$
- Pulse 15 minutes on vortex mixer
- Centrifuge for 15 minutes at 3,300 rpms with counter balance
- ✓ Make 56 AqIII (15ml), 56 OrIII Meas Vol (50 ml), 56 ORIII (50 ml), and transfer into glovebox with smaller pipette tips
- ✓ Separate phases of $56 \ AqII$, put top phase in $56 \ OrIII \ Meas \ Vol$ and bottom phase in $56 \ AqIII$
- \mathbf{Z} Separate top first, with disposable pipettes, and tried very hard to not cross contaminate anything

Friday 2, December 2016

- Went from 572 μ l to 515 μ l, what is going on?!, I didn't lose a single drop.
- ✓ Measured volume of organic by transfering from 56 OrIII Meas Vol to 56 OrIII
 - Measured a volume of 5.47 ml...how did we lose all that volume (from 5.72)!
 - I did leave maybe like 0.5 ml in 56 AqII (the interface), but still
- ${f Z}$ Take $\fbox{56~OrIII}$ and $\fbox{56~AqIII}$ out of glovebox
- Clean both vials, centrifuge 1 min 4,400 rpm
- **✓** Parafilm wrap both
- \square Start counting $\boxed{56 \ OrIII}$ at 26 cm

Saturday 3, December 2016

1 Things to do for school

☑ Worked on NUEN 647 project all day

Sunday 4, December 2016

1 Things to do for school

☑ Worked on NUEN 647 project all day

Monday 5, December 2016

1 Things to do for school

- ✓ Worked on NUEN 647 project all day
 - I know its a work day, but technically I am only getting paid for 20 hours a week.
 - Also, I shouldn't have to explain myself for doing school work...
 - Project is due Tuesday, and guess what, its not complete nor does it work

Tuesday 6, December 2016

1 Experiment to double check ¹³⁷Cs using combined aqueous series 5 and 6, 56

- \checkmark Finsih count $\boxed{56 \ OrIII}$ at 26 cm
 - The detector was being a little wonky with this, at first the data was lost, then I opened up the detector again and it was there...I hope its good?

2 Things to do for school

- ☑ Give final presentation for NUEN647
 - Should have sampled from the covariance matrix, how exactly do I do that?
 - Didn't complete the project, but its okay
 - Now all that is left is the final homework

3 Experiment to double check ¹³⁷Cs using combined aqueous series 5 and 6, 56

- - School computer (not the one making these notes) is not working. Its moving very slowly
 - Uninstalled CE, fixed it. IT people said that they installed malware software, so programs were interfering with one another
- ✓ Combine 56 OrII , 56 OrIII , 5 Or , 6 Or , 5 OrII , 6 OrII , into 56 Or .

 Stored in fume hood for now, parafilm wrap
- \mathbf{Z} Combine $\mathbf{56}$ mix, $\mathbf{56}$ mixII, $\mathbf{1}$ into $\mathbf{56}$ AqII leave in glovebox

4 Process Experiment (continuation from cycle experiment)

 \checkmark Transfer $\boxed{8 \ Or}$, $\boxed{9 \ Or}$, $\boxed{10 \ Or}$ into glovebox

☑ Combine all organic phases for each respective experiment

- \bullet [8 OrII], [8 OrII C], and [8 Or C], into [8 Or with clear push cap
- ullet $[9\ OrII], [9\ OrII\ C]$, and $[9\ Or\ C]$, into $[9\ Or]$ with clear push cap
- $\boxed{10~OrII}$, $\boxed{10~OrII~C}$, and $\boxed{10~Or~C}$, into $\boxed{10~Or}$ with clear push cap
- ✓ Combine all aqueous phases for each respective experiment
 - 8 AqII C into 8 AqII
 - $\boxed{9 \ AqII \ C}$ into $\boxed{9 \ AqII}$
- ✓ Take out trash and consolodate waste, put in new waste bag
- ☑ Clean gloves and pipette in glovebox

5 Things to Check

- □ Ru paper
- ☐ Check if using interface for second extraction interfered with results for 8,9, 10 experiment
- ✓ Wrote the procedure for tomorrow
- \mathbf{Z} Modified second extraction, which had 248 ml for $8 \ aqII \ C$

Wednesday 7, December 2016

Note...second extraction had 248 ml for $\boxed{8~aqII~C}$ change in calculations. Didn't change results much.

1 Process Experiment (continuation from cycle experiment)

Third contact

- ✓ Label vials, 8 aqIII, 8 aqIII C, 8 orIII, 8 orIII C 9 aqIII, 9 aqIII C, 9 orIII, 9 orIII C 10 aqIII, 10 aqIII C, 10 orIII, 10 orIII C, 8 aqIII Dilution 9 aqIII Dilution, 10 aqIII Dilution, 8 orIII Dilution, 9 orIII Dilution, 10 orIII Dilution (smaller 2.5 ml tubes) 8 mixIII, 9 mixIII, 10 miIIIx (smaller 1 ml tubes from John Burns, have conical bottoms, makes more minute separations easier)
- Transfer: $8 \ aqIII$, $8 \ aqIII \ C$, $8 \ orIII$, $8 \ orIII \ C$ $9 \ aqIII$, $9 \ aqIII \ C$, $9 \ aqIII \ C$, $9 \ aqIII \ C$, $10 \ aqIII \ Dilution$,
- Centrifuge $\boxed{8~aqII}$, $\boxed{9~aqII}$, $\boxed{10~aqII}$ for 3,300 rpm. 4 minutes to get liquid to bottom

Volumes for combined aqueous phases

Series	Aqueous (8,9, or 10)
8	353 + / -3%
9	353 + / -3%
10	362 + / -3%

$$\checkmark$$
 Add 362 μ l of TBP to $9 \ aqII$

• Accident!!! need to correct in calculations

- \square Vortex mix $\boxed{8 \ aqII}$ for 15 minutes on pulse mode
- ∇ Vortex mix $\boxed{9 \ aqII}$ for 15 minutes on pulse mode
- \checkmark Vortex mix $10 \ aqII$ for 15 minutes on pulse mode
- Pipette with disposable pipette the organic phase first, then the aqueous (for all three vials), (also used different disposable pipettes for the different phases no contamination) as much as so that there is no mixing. Then transferred the boundary to a smaller vial, centrifuge. Transfer rest of phases. Prepare counting solutions of $250~\mu l$ of each of the solutions A picture will be provided for the whole process for 8~aqII on the following page , also make dilution of each of the phases. Directly below are notes for what happened in the initial transfer, and below that are notes for the dilution.
 - 10 OrIII C and 10 AqIII C both have 200 μ l instead of 250 μ l. Potentially because I lost a drop of the organic phase
 - Combined all organic after making count and dilution solutions
 - Also had to recentrifuge the 10 series twice

✓ Make dilution of each aqueous

✓ -
$$\boxed{8 \ aqIII}$$
 10+/-0.3 μ l of $\boxed{8 \ aqIII}$ (4 M HNO₃) [smaller pipette] + 990+/-5.94 μ l of DI water (leftover in glovebox) = 1.0+/-0.006 ml of ~ 0 M HNO₃ $\boxed{8 \ aqIII \ Dilution}$ ✓ - $\boxed{9 \ aqIII}$ (4 M HNO₃) [smaller pipette]

10+/-0.3
$$\mu$$
l of $\boxed{9~aqIII}$ (4 M HNO₃) [smaller pipette] + 990+/-5.9 μ l of DI water (leftover in glovebox) = 1.0+/-0.006 ml of \sim 0 M HNO₃ $\boxed{9~aqIII~Dilution}$

Wednesday 7, December 2016

 \square Stop count $\boxed{8 \ AqIII \ C}$ at 5:30 pm

Wednesday 7, December 2016

Wednesday 7, December 2016



Extraction three times round 2 extraction 3

2 Details from research meeting

- Email Dr. Burns about the cheat sheet
- Use percentage that Matt used for his paper
- $\bullet\,$ Bug Dr. Folden Friday evening, maybe 1 hour of enrollment, talk to Julie Zercher look up at CHEM tamu.edu

Thursday 8, December 2016

1 Process Experiment (continuation from cycle experiment)

- \square Transfer $\boxed{8 \ AqIII \ C}$, $\boxed{9 \ AqIII \ C}$, $\boxed{10 \ AqIII \ C}$ into glove box
 - In plastic bag (2.5 ml in a 15 ml parafilm wrapped), inside the glass beaker the plastic bag popped under the negative pressure and the glass beaker tipped over
 - Centrifuged vials at 2500 rpm for 4 minutes

Fourth contact

- Transfer: $8 \ aqIII$, $8 \ aqIII \ C$, $8 \ orIII$, $8 \ orIII \ C$ $9 \ aqIII$, $9 \ aqIII \ C$, $9 \ aqIII \ C$, $10 \ aqIIII \ C$, $10 \ aqIII \$
- \mathbf{Z} Transfer $abla AqIII C + \underline{8 \ AqIII}$ (old) into $abla 8 \ AqIII$ (new)
- \square Transfer $\boxed{9 \ AqIII \ C}$ into $\boxed{9 \ AqIII}$ (old) into $\boxed{9 \ AqIII}$ (new)
- \mathbf{Z} Transfer $\boxed{10 \ AqIII \ C}$ into $\boxed{10 \ AqIII}$ (old) into $\boxed{10 \ AqIII}$ (new)

Thursday 8, December 2016

Volumes for combined aqueous phases

Series	Aqueous (8,9, or 10)
8	315 + / -3%
9	320 + /-3%
10	308 + / -3%

- \checkmark Vortex mix $\boxed{8 \ aqIII}$ for 15 minutes on pulse mode
- \checkmark Vortex mix $\boxed{9 \ aqIII}$ for 15 minutes on pulse mode
- ✓ Vortex mix 10 aqIII for 15 minutes on pulse mode
- \mathbf{Z} Centrifuge $\boxed{8 \ aqIII}$, $\boxed{9 \ aqIII}$, and $\boxed{10 \ aqIII}$ with \boxed{Buddy} on 3300 rpm, for 10 minutes
- Pipette with disposable pipette the organic phase first, then the aqueous (for all three vials), (also used different disposable pipettes for the different phases no contamination) as much as so that there is no mixing. Then transferred the boundary to a smaller vial, centrifuge, then transfer the rest. Prepare counting solutions of 250 μ l of each of the solutions A picture will be provided for the whole process for $8 \ aqIII$ on the following page, also make dilution of each of the phases. Directly below are notes for what happened in the initial transfer, and below that are notes for the dilution. Also consolodated waste after experiment. All interfaces into the first mix $8 \ Mix$, $9 \ mix$, and $10 \ mix$, also all excess aqueous phase was put into here as well. All excess organic (after dilution and creation of count vial, was put into a single organic vial $8 \ Or$, $9 \ Or$, and $10 \ Or$.)
 - When transfering interface of 8 AqIII lost a drop
 - Also $\fbox{8~OrIV~C}$ might have 248 μl (less than 250)

• Same with
$$9 OrIV C$$

10+/-0.3
$$\mu$$
l of $\boxed{8~aqIV}$ (4 M HNO₃) [smaller pipette] + 990+/-5.94 μ l of DI water (leftover in glovebox) = 1.0+/-0.006 ml of \sim 0 M HNO₃ $\boxed{8~aqIV~Dilution}$

10+/-0.3
$$\mu$$
l of $\boxed{9~aqIV}$ (4 M HNO₃) [smaller pipette] + 990+/-5.9 μ l of DI water (leftover in glovebox) = 1.0+/-0.006 ml of \sim 0 M HNO₃ $\boxed{9~aqIV~Dilution}$

10+/-0.3
$$\mu$$
l of $10~aqIV$ (4 M HNO₃) [smaller pipette] + 990+/-7.8 μ l of DI water (leftover in glovebox) = 1.0+/-0.006 ml of \sim 0 M HNO₃ $10~aqIV~Dilution$

✓ Make dilution of each organic phase

10+/-0.3
$$\mu$$
l of $\boxed{8~orIV}$ (30% TBP) [smaller pipette] + 990+/-7.8 μ l of 30% TBP (leftover in glovebox) = 1.0+/-0.006 ml of 30% TBP $\boxed{8~orIV~Dilution}$

10+/-0.3
$$\mu$$
l of $\boxed{9~orIV}$ (30% TBP) [smaller pipette] + 990+/-7.8 μ l of 30% TBP (leftover in glovebox) = 1.0+/-0.006 ml of 30% TBP $\boxed{9~orIV~Dilution}$

Thursday 8, December 2016

10+/-0.3
$$\mu$$
l of 10 $orIV$ (30% TBP) [smaller pipette] + 990+/-7.8 μ l of 30% TBP (leftover in glovebox) = 1.0+/-0.006 ml of 30% TBP 10 $orIV$ $Dilution$

- $lefont{rac{1}{2}}$ Transfer out $lefont{8 \ orIV \ C}$, $lefont{8 \ aqIV \ C}$, $lefont{9 \ orIV \ C}$, $lefont{9 \ aqIV \ C}$, $lefont{10 \ orIV \ C}$, $lefont{10 \ aqIV \ C}$, in 15 ml centrifuge tubes
- ✓ Clean stuff in glovebox
- ${f f Z}$ Radiac wash the above tubes, and store in fumehood behind lead wait to count
- ✓ Centrifuge 8 or IV C, 8 aq IV C, 9 or IV C, 9 aq IV C, 10 or IV C, 10 aq IV C, for 2 minutes 4,400 rpm to put all liquid at bottom, then parafilm wrap all the vials and store in fumehood.

Thursday 8, December 2016



Extraction three times round 2 extraction 3

2 Things to Check

 \Box Dr. Burns mentioned that Pu-238 overlaps with $^{241}\mathrm{Am}$ in the alpha spec

Friday 9, December 2016

1 Process Experiment (continuation from cycle experiment)

```
{\bf { \vec { V}}} Stop count \boxed{9~OrIII~C}~(\sim~7{:}20~{\rm am})
```

 ${\bf \not\!\! C}$ Start count ${\bf \boxed{8~OrIV~C}}$ (late afternoon)

- \square Dr. McClarren's homework
 - This homework...is so long

Satuday 10, December 2016

1 Process Experiment (continuation from cycle experiment)

- □ Dr. McClarren's Homework
 - This homework...is so long

Sunday 11, December 2016

1 Process Experiment (continuation from cycle experiment)

- $\hfill\Box$ Dr. McClarren's Homework
 - This homework...is so long

Monday 12, December 2016

- \square Dr. McClarren's Homework
 - This homework...is so long

Tuesday 13, December 2016

- \square Dr. McClarren's Homework
 - $\bullet\,$ This homework... is so long

Wednesday 14, December 2016

1 Things to do for school

☑ Dr. McClarren's Homework

- This homework...is so long
- But finally finished, it was 62 pages...

Thursday 15, December 2016

Prepare for back extraction... Combine all aqueous,

- Transfer $\boxed{8 \ OrIII \ C}$, $\boxed{9 \ OrIII \ C}$, $\boxed{10 \ OrIII \ C}$, $\boxed{8 \ AqIV \ C}$, $\boxed{9 \ AqIV \ C}$, $\boxed{10 \ AqIV \ C}$, $\boxed{8 \ OrIV \ C}$, $\boxed{10 \ OrIV \ C}$ in plastic bag into the glove box

- - $10 \ Mix$ should be centrifuged next time used

Combine all organic...

Create dilution of combined total organic

$$m{\mathbb{Z}}$$
 Transfer $m{8}$ Or Tot Dilution, $m{9}$ Or Tot Dilution, and $m{10}$ Or Tot Dilution, into glovebox

✓ Make dilution of each organic phase

Thursday 15, December 2016

10+/-0.3
$$\mu$$
l of $\boxed{8\ or}$ (30% TBP) [smaller pipette] + 990+/-7.8 μ l DI water (leftover in glovebox) = 1.0+/-0.006 ml of DI Water $\boxed{8\ or\ Tot\ Dilution}$

✓ - $\boxed{9\ or}$

10+/-0.3 μ l of $\boxed{9\ or}$ (30% TBP) [smaller pipette] + 990+/-7.8 μ l of DI water (leftover in glovebox) = 1.0+/-0.006 ml of DI water $\boxed{9\ or\ Tot\ Dilution}$

✓ - $\boxed{10\ or}$

10+/-0.3 μ l of $\boxed{10\ or}$ (30% TBP) [smaller pipette] + 990+/-7.8 μ l of DI water (leftover in glovebox) = 1.0+/-0.006 ml of DI water (leftover in glovebox) = 1.0+/-0.006 ml of DI water $\boxed{10\ or\ Tot\ Dilution}$

Measure volume of organic

done Centrifuge 5 minutes for 2500 rpm 8 or , 9 or , and 10 or with buddy

Volumes for combined organic phases

Series	Organic (8,9, or 10)
8	888
9	912
10	863

That is much less than what I expected, if this were a perfect world, there should be $1600 \mu l$ in those vials.

Count the organic phases

- ✓ Transfer red lids into glovebox
- ✓ Change clear to red lids on the organic phases
- ${f Z}$ Transfer out ${f 8}$ or , ${f 9}$ or , ${f 10}$ or glovebox, clean, parafilm wrap, centrifuge

Thursday 15, December 2016

✓ Start count 8 Or at 0 cm around 11:00 am, there is a 5% dead time, which is about what was expected
✓ Stop count 8 Or at 0 cm (2:20 pm)
✓ Start count 9 Or at 0 cm (2:20 pm)
✓ Stop count 9 Or at 0 cm (5:20 pm)
✓ Start count 10 Or at 0 cm (5:20 pm) - leave over night

Prepare for the next few days

- ✓ Write procedure for back extractions
- \square Analyze results from previous extractions
 - ✓ Extraction 3, extraction 4
 - reanalyze alpha results with what Dr. Burns mentioned, write script to minimize variance of results varying parameters like volume (not really)
- ✓ Look at homework for 629

Friday 16, December 2016

1 Process Experiment (continuation from cycle experiment)

Counting

$$\checkmark$$
 Transfer $\boxed{8 \ Or}$, $\boxed{9 \ Or}$, and $\boxed{10 \ Or}$ into glove box

Back extraction experiment 1. Note \boxed{B} is for back extraction. Also special vials labeled below are the vials with clear push caps that actually make a good seal.

Label vials,
$$8 \ aqB$$
, $8 \ aqB \ C$, $8 \ orB$ (special) $9 \ orB \ C$ $9 \ aqB$, $9 \ aqB \ C$, $9 \ orB$ (special), $9 \ orB \ C$ $10 \ aqB$, $10 \ aqB \ C$, $10 \ orB$ (special), $10 \ orB \ C$, $10 \ orB \ Dilution$, $10 \ aqB \ Dilution$, $10 \ aqB \ Dilution$, $10 \ orB \ Dilu$

Transfer:
$$8 \ aqB$$
, $8 \ aqB \ C$, $8 \ orB$ (special) \odot , $8 \ orB \ C$ $9 \ aqB$, $9 \ aqB \ C$, $9 \ orB \ C$ $10 \ aqB$, $10 \ aqB \ C$, $10 \ orB \ Dilution$, $10 \$

Volumes for combined organic phases

Series	Organic (8,9, or 10)
8	888
9	912
10	863

Create an Fe solution for a back extraction, $Fe\ Prep$ (Small portions created right before the experiment because this solution has a short half life with larger concentrations of HNO_3).

$$0.0417+/-0.0018 \text{ ml of } 2.302+/-0.009 \text{ M Fe(II) in } 0.0+/-0 \text{ M HNO}_3$$
 $Stock \ Fe(II)$ $+$ $3.941+/-0.027 \text{ ml of } 0.0+/-0 \text{ M Fe(II) in } 4.06+/-0.05 \text{ M HNO}_3 \text{ solution } Fe \ Prep$ $+$ $4.000+/-0.020 \text{ ml of } 0.0240+/-0.0010 \text{ M Fe(II) in } 4.00+/-0.05 \text{ M HNO}_3 \text{ solution } \longrightarrow Bk \ Ex \ Solution$.

Actual Back extraction

- ✓ Vortex mix 8 or for 15 minutes on pulse mode
- ✓ Vortex mix 9 or for 15 minutes on pulse mode
- $\mathbf{\nabla}$ Vortex mix $\boxed{10 \text{ or}}$ for 15 minutes on pulse mode
- Pipette with disposable pipette the organic phase first, then the aqueous (for all three vials), (also used different disposable pipettes for the different phases no contamination) as much as so that there is no mixing. Then transferred the boundary to a smaller vial, centrifuge (3,300 for ~ 5 minutes), then transfer the rest. Prepare counting solutions of 500 μ l of each of the solutions A picture will be provided for the whole process for some step for series 8, on the following page (picture is of a previous experiment, but same process), also make dilution of each of the phases. Directly below are notes for what happened in the initial transfer, and below that are notes for the dilution.
 - Did pretty well this time around
- ✓ Make dilution of each aqueous

10+/-0.3
$$\mu$$
l of $\boxed{8~aqB}$ (4 M HNO₃) [smaller pipette] + 990+/-5.94 μ l of DI water (leftover in glovebox) = 1.0+/-0.006 ml of \sim 0 M HNO₃ $\boxed{8~aqB~Dilution}$

Friday 16, December 2016



Back extraction I, (different vial names, same procedure)

✓ Write up procedure for weekend and Monday

2 Things we need to buy

I kind of don't want to ask for this because anytime money comes up with Dr. Chirayath he gets really rude and mean.

 \square 500, 15 ml centrifuge tubes from VWR, catalog # 89401-574 priced at \$302.98

3 Process Experiment (continuation from cycle experiment)

- \checkmark Start count $\boxed{9 \ OrB \ C}$ at 12:36 am

Saturday 17, December 2016

1 Process Experiment (continuation from cycle experiment)

Counting

Sunday 18, December 2016

1 Process Experiment (continuation from cycle experiment)

Counting

- ${\bf { \vec Z}}$ Start count $\overline{ \left[8~AqB~C \right] }$ at 8:00 am
- \mathbf{Z} Stop count abla AqB C at 5:00 pm
- \mathbf{Z} Stop count $\boxed{9 \text{ } AqB \text{ } C}$ at 10:00 pm

Monday 19, December 2016

1 Process Experiment (continuation from cycle experiment)

Counting

- \square Start count $\boxed{9 \ OrBII \ C}$ at 11:00 am

Prepare for second back extraction

- $\ensuremath{\square}$ Transfer into glovebox $\ensuremath{8~orB~C}$, $\ensuremath{9~orB~C}$, $\ensuremath{10~orB~C}$, in 15 ml centrifuge tubes, parafilm wrapped, in plastic bag
- Label vials, $[8 \ aqBII]$, $[8 \ aqBII \ C]$, $[8 \ orBII]$ (special) $[9 \ aqBII \ C]$, $[9 \ aqBII \ C]$, $[9 \ aqBII \ C]$, $[9 \ orBII \ C]$, $[10 \ aqBII \ Dilution]$, [
- Transfer: $8 \ aqBII$, $8 \ aqBII \ C$, $8 \ orBII$ (special) \odot , $8 \ orBII \ C$ $9 \ aqBII \ C$, $9 \ orBII \ C$, $9 \ orBII \ C$, $9 \ orBII \ C$, $10 \ aqBII \ C$, $10 \ aqBII \ C$, $10 \ orBII \ Dilution$, $10 \ aqBII \ Diluti$

Combine all organic phases

Combine all aqueous phases (still counting)

 \square Transfer 8 AqB C into 8 AqB

Monday 19, December 2016

- \square Transfer $9 \ AqB \ C$ into $9 \ AqB$
- \square Transfer $10 \ AqB \ C$ into $10 \ AqB$

Measure volume of organic phases

- ☑ Centrifuge 8 OrB, 9 OrB, 10 OrB for 2,500 rpm. 4 minutes to get liquid to bottom
- \square Measure volumes of all organic phases, $\boxed{8 \ OrB} \boxed{9 \ OrB}$, $\boxed{10 \ OrB}$

Volumes for combined organic phases

Series	Organic (8,9, or 10)
8	760
9	765
10	770

Create an Fe solution for a back extraction, $Fe\ Prep$ (Small portions created right before the experiment because this solution has a short half life with larger concentrations of HNO_3).

✓ -

$$0.0417+/-0.0018 \text{ ml of } 2.302+/-0.009 \text{ M Fe(II) in } 0.0+/-0 \text{ M HNO}_3$$
 $Stock \ Fe(II)$ $+$ $3.941+/-0.027 \text{ ml of } 0.0+/-0 \text{ M Fe(II) in } 4.06+/-0.05 \text{ M HNO}_3 \text{ solution } Fe \ Prep$ $+$ $4.000+/-0.020 \text{ ml of } 0.0240+/-0.0010 \text{ M Fe(II) in } 4.00+/-0.05 \text{ M HNO}_3 \text{ solution } \longrightarrow Bk \ Ex \ Solution$.

Actual Back extraction

- \checkmark Vortex mix 8 orB for 15 minutes on pulse mode
- ∇ Vortex mix 9 orB for 15 minutes on pulse mode
- \checkmark Vortex mix 10 orB for 15 minutes on pulse mode

- Pipette with disposable pipette the organic phase first, then the aqueous (for all three vials), (also used different disposable pipettes for the different phases no contamination) as much as so that there is no mixing. Then transferred the boundary to a smaller vial, centrifuge, then transfer the rest. Prepare counting solutions of 500 μ l of each of the solutions A picture will be provided for the whole process for some step for series 8, on the following page (picture is of a previous experiment, but same process), also make dilution of each of the phases. Directly below are notes for what happened in the initial transfer, and below that are notes for the dilution. Also after dilution and counting solutions are made, combine all remaining aqueous solution into $8 \ aqB$, $9 \ aqB$, or $10 \ aqB$.
 - 10~AqBII~C probably doesn't have 500 μ l maybe 480-495
- ✓ Make dilution of each aqueous

990+/-5.94 μ l of DI water (leftover in glovebox) =

1.0+/-0.006 ml of
$$\sim 0$$
 M HNO₃ $\boxed{8~aqBII~Dilution}$

$$10+/\text{-}0.3~\mu\text{l}$$
 of $\boxed{9~aqBII}$ (4 M HNO3) [smaller pipette] $+$ 990+/-5.9 μl of DI water (leftover in glovebox)

$$1.0 + /\text{-}0.006 \text{ ml of} \sim 0 \text{ M HNO}_3$$
 9 aqBII Dilution

10+/-0.3
$$\mu$$
l of 10 $aqBII$ (4 M HNO₃) [smaller pipette] + 990+/-7.8 μ l of DI water (leftover in glovebox)

1.0+/-0.006 ml of
$$\sim$$
 0 M HNO₃ 10 aqBII Dilution

✓ Make dilution of each organic phase

Monday 19, December 2016

$$10+/\text{-}0.3~\mu\text{l of }\boxed{8~orBII}~(30\%~\text{TBP})~[\text{smaller pipette}]\\ +\\ 990+/\text{-}7.8~\mu\text{l of DI water (leftover in glovebox)}\\ =\\ 1.0+/\text{-}0.006~\text{ml of DI water }\boxed{8~orBII~Dilution}$$
 \$\mathbf{\sigma}\$ - \begin{array} 9~orBII \\ 10+/\text{-}0.3~\mu\text{l of }\begin{array} 9~orBII \\ 10+/\text{-}0.3~\mu\text{

+/-0.3 μ l of 9 orBII (30% TBP) [smaller pipette] + 990+/-7.8 μ l of DI water (leftover in glovebox) = 1.0+/-0.006 ml of DI water 9 orBII Dilution

☑ - 10 orBII

10+/-0.3 μ l of 10 orBII (30% TBP) [smaller pipette] + 990+/-7.8 μ l of DI water (leftover in glovebox) = 1.0+/-0.006 ml of DI water 10 orBII Dilution

- Transfer out $\boxed{8 \ or BII \ C}$, $\boxed{8 \ aqBII \ C}$, $\boxed{9 \ or BII \ C}$, $\boxed{9 \ aqBII \ C}$, $\boxed{10 \ or BII \ C}$, in 15 ml centrifuge tubes
- \square Clean stuff in glovebox
- ✓ Centrifuge 8 or BII C, 8 aqBII C, 9 or BII C, 9 aqBII C, 10 or BII C, 10 aqBII C, for 2 minutes 4,400 rpm to put all liquid at bottom, then parafilm wrap all the vials and store in fumehood.

Counting

- \mathbf{Z} Start count $\boxed{10 \text{ } OrBII \text{ } C}$ around 10:00pm

Monday 19, December 2016



Back extraction I, (different vial names, same procedure)

Tuesday 20, December 2016

1 Process Experiment (continuation from cycle experiment)

Counting

- \square Start count for $\boxed{10~AqB~C}$ (finish out back extraction aqueous counts)

Prepare for third back extraction

- Label vials, $8 \ aqBIII$, $8 \ aqBIII \ C$, $8 \ orBIII$ (special) \odot , $8 \ orBIII \ C$ $9 \ aqBIII \ C$, $9 \ aqBIII \ C$, $9 \ orBIII \ C$, $9 \ orBIII \ C$, $10 \ aqBIII \ C$, $10 \ aqBIII \ C$, $10 \ aqBIII \ Dilution$, $10 \ orBIII \ Dilution$, 1
- Transfer: $8 \ aqBIII$, $8 \ aqBIII \ C$, $8 \ orBIII$ (special) \odot , $8 \ orBIII \ C$ $9 \ aqBIII \ C$, $9 \ aqBIII \ C$, $9 \ orBIII \ C$, $9 \ orBIII \ C$, $10 \ aqBIII \ Dilution$, $10 \ orBIII \ Dilution$, $10 \ orBIII$
- ${f Z}$ Transfer in disposable pipettes and small pipettes

Combine all organic phases

Combine all aqueous phases (still counting them)

- \square Transfer $\boxed{8 \ AqBII \ C}$ into $\boxed{8 \ AqBII}$
- \square Transfer 9 AqBII C into 9 AqBII
- \Box Transfer $\boxed{10~AqBII~C}$ into $\boxed{10~AqBII}$

Measure volume of organic phases

Volumes for combined organic phases

Series	Organic (8,9, or 10)
8	618
9	620
10	621

Create an Fe solution for a back extraction, $Fe\ Prep$ (Small portions created right before the experiment because this solution has a short half life with larger concentrations of HNO_3).

√ _

$$0.0417+/-0.0018 \text{ ml of } 2.302+/-0.009 \text{ M Fe(II) in } 0.0+/-0 \text{ M HNO}_3$$
 $Stock\ Fe(II)$ $+$ $3.941+/-0.027 \text{ ml of } 0.0+/-0 \text{ M Fe(II) in } 4.06+/-0.05 \text{ M HNO}_3 \text{ solution }$ $Fe\ Prep$ $+$ $4.000+/-0.020 \text{ ml of } 0.0240+/-0.0010 \text{ M Fe(II) in } 4.00+/-0.05 \text{ M HNO}_3 \text{ solution }$ $\rightarrow Bk\ Ex\ Solution$ $.$

Actual Back extraction

- \checkmark Vortex mix $\boxed{8 \text{ } orBII}$ for 15 minutes on pulse mode
- \checkmark Vortex mix $\boxed{9 \ or BII}$ for 15 minutes on pulse mode

- \checkmark Vortex mix 10 orBII for 15 minutes on pulse mode
- Pipette with disposable pipette the organic phase first, then the aqueous (for all three vials), (also used different disposable pipettes for the different phases no contamination) as much as so that there is no mixing. Then transferred the boundary to a smaller vial, centrifuge, then transfer the rest. Prepare counting solutions of 400 μ l of each of the solutions A picture will be provided for the whole process for some step for series 8, on the following page (picture is of a previous experiment, but same process), also make dilution of each of the phases. Directly below are notes for what happened in the initial transfer, and below that are notes for the dilution. Also after dilution and counting solutions are made, combine all remaining aqueous solution into 8~aqB, 9~aqB, or 10~aqB.
 - Dropped $10 \ OrIIB$ (after initial centi, so centrifuged again for 5 minutes at 2500 rpm)
 - Interfece of 8 series dropped. fell on paper towels after the incident transfered all interfaces into a single mix smaller vial
- ✓ Make dilution of each aqueous

✓ -
$$\boxed{8\ aqBIII}$$

10+/-0.3 μ l of $\boxed{8\ aqBIII}$ (4 M HNO₃) [smaller pipette] +

990+/-5.94 μ l of DI water (leftover in glovebox)

=
1.0+/-0.006 ml of ~ 0 M HNO₃ $\boxed{8\ aqBIII\ Dilution}$

✓ - $\boxed{9\ aqBIII}$

10+/-0.3 μ l of $\boxed{9\ aqBIII}$ (4 M HNO₃) [smaller pipette] +

990+/-5.9 μ l of DI water (leftover in glovebox)

=
1.0+/-0.006 ml of ~ 0 M HNO₃ $\boxed{9\ aqBIII\ Dilution}$

✓ - $\boxed{10\ aqBIII}$

10+/-0.3 μ l of $\boxed{10\ aqBIII}$ (4 M HNO₃) [smaller pipette] +

990+/-7.8 μ l of DI water (leftover in glovebox)

=
1.0+/-0.006 ml of ~ 0 M HNO₃ $\boxed{10\ aqBIII\ Dilution}$

✓ Make dilution of each organic phase

$$10+/\text{-}0.3~\mu\text{l}$$
 of 8 $or BIII$ (30% TBP) [smaller pipette] $+$ 990+/-7.8 μl of DI water (leftover in glovebox)

= 1.0 + /-0.006 ml of DI water 8 or BIII Dilution

10+/-0.3
$$\mu$$
l of $\boxed{9~orBIII}$ (30% TBP) [smaller pipette] + 990+/-7.8 μ l of DI water (leftover in glovebox) =

1.0+/-0.006 ml of DI water $\boxed{9~or BIII~Dilution}$

$$10+/-0.3~\mu l$$
 of $10~orBIII$ (30% TBP) [smaller pipette] + $990+/-7.8~\mu l$ of DI water (leftover in glovebox) = $1.0+/-0.006~m l$ of DI water $10~orBIII~Dilution$

- ✓ Transfer out $\boxed{8 \ or BIII \ C}$, $\boxed{8 \ aqBIII \ C}$, $\boxed{9 \ or BIII \ C}$, $\boxed{9 \ aqBIII \ C}$, $\boxed{10 \ aqBIII \ C}$, in 15 ml centrifuge tubes
- ✓ Clean stuff in glovebox
- \square Radiac wash the above tubes, and store in fumehood behind lead wait to count
- ✓ Centrifuge 8 or BIII C, 8 aqBIII C, 9 or BIII C, 9 aqBIII C, 10 or BIII C, 10 aqBIII C, for 2 minutes 4,400 rpm to put all liquid at bottom, then parafilm wrap all the vials and store in fumehood.

Counting

- ✓ Start count 8 OrBIII C

Tuesday 20, December 2016



Back extraction I, (different vial names, same procedure)

Wednesday 21 December 2016, to January 5th, 2017

1 Process Experiment (continuation from cycle experiment)

Counting

Counting of all the samples. There were lots of aqueous samples to count. During this time I also visited family. I completed my incomplete for NUEN629, which came about because of a shattered kneecap.

Wednesday January 18th, 2017

1 Process Experiment (continuation from cycle experiment)

Analyzed data for experiments. Went to lab meeting and left kind of angry, went home to cool off.

Thursday January 19th, 2017

1 Prepare Alpha chips for counting, the mass spec solutions for shipping, count the final combined aqueous phases

Make chips...
Transfer all samples to twist cap
Combine final aqueous
Count Final aqueous
Analyze final aqueous
Finish proposal

2 Work that is paying me this semester

Attend Lab trainning.