Laboratory Journal

Paul Mendoza

paul.m.mendoza@gmail.com

This notebook begins 6 October 2016

Contents

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

1 Isotopes we are looking for

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

^{133}Cs	^{136}Ba	^{153}Eu
^{134}Cs	^{138}Ba	^{154}Eu
^{135}Cs	^{149}Sm	^{239}Pu
^{137}Cs	^{150}Sm	^{242}Pu

Table 1: 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
- ^{152}Eu Liquid calibration source
  - Source 1577-22
  - 497.0 nCi
  - Assy Date: 15 Feb 12
  - 1.00568g
- Stock HNO_3: 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 1000μl pipett from glovebox
- Decontaminate with radic - dump waste into glass aq rad outside glove box
- Practice pipetting 500μl to glass vial - setting 503 μl gives 500 μl
- 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:

$$M_2 = \frac{M_3V_3 - M_1V_1}{V_3 - V_1}$$
$$V_2 = V_3 - V_1$$

Use to determine molality $\rightarrow wt\%_2 \rightarrow \rho_2$. 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

☑ Prepare shielding for transfer for closet solution

- 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

☑ -

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

☑ -

$$\text{Combine } 0.500\text{+/-}0.005 \text{ ml of } 15.43\text{+/-}0.06 \text{ M } HNO_3 \text{ solution } \boxed{closet}$$
$$+$$
$$2.048\text{+/-}0.026 \text{ ml of } 1.12\text{+/-}0.08 \text{ M } HNO_3 \text{ solution } \boxed{Stock}$$
$$=$$
$$2.500\text{+/-}0.025 \text{ ml of } 4.00\text{+/-}0.05 \text{ M } HNO_3 \text{ solution. } \boxed{\rightarrow Stock}$$

☑ Lock $\boxed{Stock}$ in glovebox

☑ 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

☑ Start background count and done for the day

- Count lasted for 12 hours

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

1 Preparation for Process 1

- ☑ Finish background count, lasted 12 hours
- ☑ Remove 0.3 ml from $\boxed{Stock}$ transfer to $\boxed{1}$ for counting
  - $\boxed{1}$ is a smaller tube, which will fit into a larger centrifuge tube for, well, centrifuging
  - $\boxed{1}$ 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
  - 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
- ☑ Count $\boxed{1}$ for 1 hour and 24 minutes
- ☑ Fix density calculation in code, was slightly wrong before, this means $\boxed{Stock}$ and $\boxed{1}$ are slightly different from what they should be, but within error
- ☑ Calculation for creation of Fe(II) solution (next page)

$$
\begin{gathered}
V_1 \text{ ml of } M_{1,Fe} \text{ Fe(II) in } M_{1,HNO_3} \text{ HNO}_3 \\
+ \\
V_2 \text{ ml of } M_{2,Fe} \text{ Fe(II) in } M_{2,HNO_3} \text{ HNO}_3 \\
= \\
V_3 \text{ ml of } M_{3,Fe} \text{ Fe(II) in } M_{3,HNO_3} \text{ HNO}_3.
\end{gathered}
$$

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 HNO_3 constant: $V_2 = \frac{V_3 M_{3,HNO_3}}{M_{2,HNO_3}}$
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 HNO_3 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 $\boxed{Stock}$ 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

☑ -

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

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

☐ -

0.0417+/-0.0018 ml of 2.302+/-0.009 M Fe(II) in 0.0+/-0 M HNO_3 $\boxed{Stock\ Fe(II)}$
+
3.941+/-0.027 ml of 0.0+/-0 M Fe(II) in 4.06+/-0.05 M HNO_3 solution $\boxed{Fe\ Prep}$
+
4.000+/-0.020 ml of 0.0240+/-0.0010 M Fe(II) in 4.00+/-0.05 M HNO_3 solution
$\boxed{\rightarrow Bk\ Ex\ Solution}$.

☑ Add Sodium Nitrite to $\boxed{1}$, it will sit overnight, but it doesn't have to

- Dropped $\boxed{1}$, 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 ml $\boxed{TBP}$ to $\boxed{1}$
- Shake on Pulse Mode of 15 minutes on vortex mixer
- Change of plans (This occured 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 $\boxed{Vial\ 1\ Budd}$ and $\boxed{Vial\ 2\ Budd}$ 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 $\boxed{TBP}$ to $\boxed{1}$ (for 0.2 ml buffer)
  - All extractions at once (different from original exp)

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

  - 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) $\boxed{\rightarrow 2}$

☑ Creation of $\boxed{Bk\ Ex\ Solution}$

0.0417+/-0.0018 ml of 2.302+/-0.009 M Fe(II) in 0.0+/-0 M HNO_3 $\boxed{Stock\ Fe(II)}$
+
3.941+/-0.027 ml of 0.0+/-0 M Fe(II) in 4.06+/-0.05 M HNO_3 solution $\boxed{Fe\ Prep}$
+
4.000+/-0.020 ml of 0.0240+/-0.0010 M Fe(II) in 4.00+/-0.05 M HNO_3 solution
$\boxed{\rightarrow Bk\ Ex\ Solution}$.

☑ Back Extraction - First Contact

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

☑ Back Extraction - Second Contact

- Add 1.2 $\boxed{Bk\ Ex\ Solution}$ to $\boxed{2}$
- Shake pulse mode for 15 minutes
- Remove 1.2 ml of bottom phase (Fe(II)) $\boxed{\rightarrow 3}$

☑ Back Extraction - Third Contact

- Add 1.2 $\boxed{Bk\ Ex\ Solution}$ to $\boxed{2}$
- Shake pulse mode for 15 minutes
- Remove 1.2 ml of bottom phase (Fe(II)) $\boxed{\rightarrow 3}$

This experiment had sputtering of pipette at certain times.

2 Counting for Process 1 Mistake experiment (Gamma)

☑ Counted waste $\boxed{1}$, containing 0.3 ml of $\boxed{Stock}$ and 0.2 ml $\boxed{TBP}$, on HPGe, for 12 hours (left for the night)

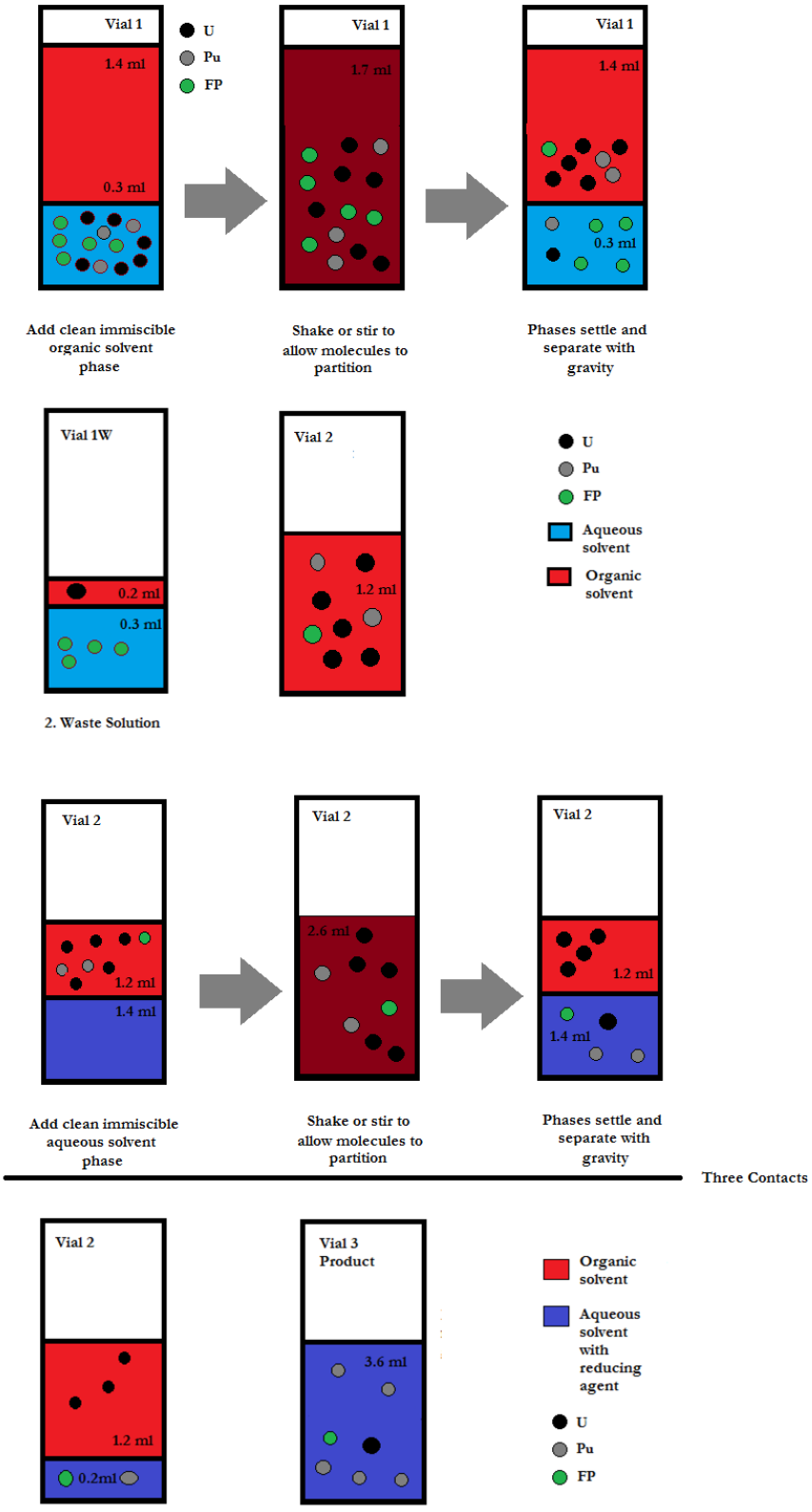

Figure 1: Process 1 Mess Up Experimental Overview

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

1 Counting for Process 1 Mistake experiment (Gamma)

There are 6 things to count.

- ☑ Initial solution $\boxed{1}$ - 23 cm away, 0.3 ml HNO_3
- ☑ Waste $\boxed{1}$ - 23 cm away, 0.3 ml HNO_3 0.2 ml TBP
- ☑ Create 4 M HNO_3 solution store in fume hood

  2.6056+/-0.0026 ml of 15.35+/-0.13 M HNO_3 solution $\boxed{Stock\ HNO_3}$
  +
  7.625+/-0.008 ml of 0.0+/-0 M HNO_3 solution $\boxed{DI}$
  +
  9.985+/-0.035 ml of 4.01+/-0.04 M HNO_3 solution $\boxed{\rightarrow 4\ M\ HNO_3}$.

- ☑ Pull out 0.2 from bottom of $\boxed{1}$ (HNO_3), dillute to 0.3 ml with $\boxed{4\ M\ HNO_3}$ $\boxed{\rightarrow 1W}$ (Part)
  - Count on HPGe $\sim$ 1 hour
- ☑ Pull out 0.3 ml from $\boxed{3}$ to count $\boxed{\rightarrow 3P}$ (product)
  - Start Count on HPGe 4 hours (left overnight)
- ☑ Pull out 0.3 ml from top of $\boxed{2}$ (TBP), to count $\boxed{\rightarrow 2W}$ (Waste)
- ☐ ~~Pull out 0.7 ml from top of $\boxed{2}$ (TBP) $\boxed{\rightarrow 2W2}$, then count $\boxed{2}$ - which should have 0.3 ml, 0.1 ml of TBP, and 0.2 ml of HNO_3~~
  - Coult not pull out all 0.7, but only 0.6
- ☑ Pull out 0.6 ml from top of $\boxed{2}$ (TBP) $\boxed{\rightarrow 2W2}$, should have 0.5 ml, 0.3 ml of TBP, and 0.2 ml of HNO_3

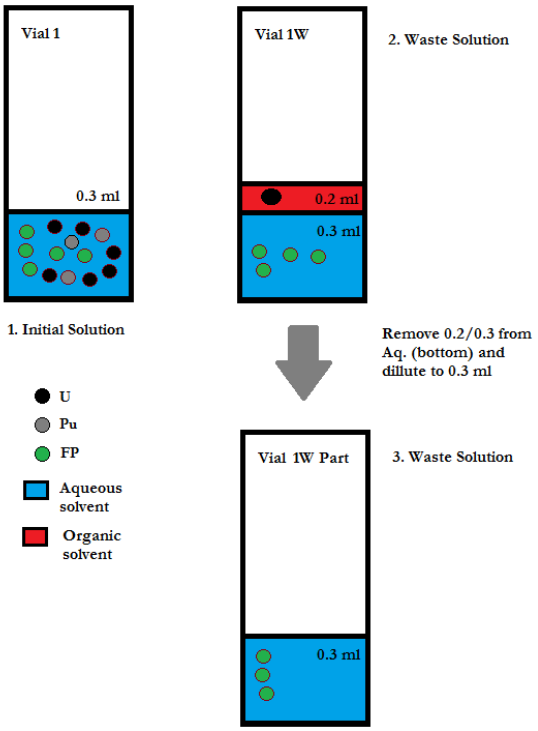

Figure 1: First Three Counts

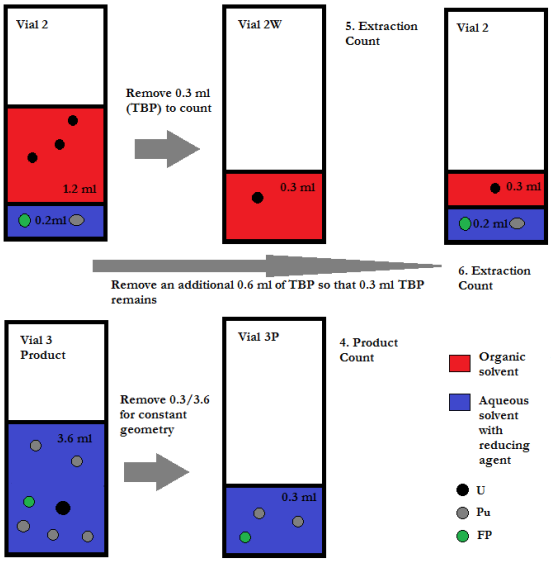

Figure 2: Second Three Counts

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

1 Counting for Process 1 Mistake experiment (Gamma)

☑ Finish count $\boxed{3P}$

☑ Start sample $\boxed{2W}$

☑ Determine preliminary results

- Determined ^{137}Cs, ^{144}Ce, ^{106}Rh 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

- Concentrated stock by a factor of two
- Decreased initial volume
- Have to mainatin, 0.2 ml excess volume to pipette from top
- Have to maintain, 0.1 ml excess from bottom
- 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 $\boxed{2W}$

2 Counting for Process 1 Mistake experiment (Alpha)

☑ Start count $\boxed{2}$

☑ 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

☑ Counted Alpha Calibration (9 mm position)

☑ Prepare alpha sample of $\boxed{Stock}$

- From Jarrod's stock 10μl was dilluted to 1ml and 10 μl was taken

10 μl of $\boxed{\textit{Stock}}$ (4 M HNO_3)
+
~~190 μl of DI water (leftover in glovebox)~~
990 μl of DI water (leftover in glovebox)
=
~~0.2 ml of $\sim$ 0 M HNO_3~~ ~~$\boxed{\textit{4 Dillution}}$~~
1 ml of $\sim$ 0 M HNO_3 $\rightarrow$ $\boxed{\textit{4 Dillution}}$

☑ Prepare and count alpha sample of Stock

- Take 20 μl of $\boxed{\textit{4 Dillution}}$, 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

☑ Count $\boxed{D1}$ over night

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

1 Counting for Process 1 Mistake experiment (Gamma)

☑ Finish count $\boxed{2}$

2 Counting for Process 1 Mistake experiment (Alpha)

☑ Finish count for $\boxed{D1}$

☑ Move $\boxed{D1}$ to safe (or glovebox)

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)

☐ Looked into alpha calibration math some more

☑ Analyze and automate (somewhat) Gamma analysis

- Program for pulling peak data from GENIE
- Program for calculating efficency 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 μl and taking out 350 μl and then getting as much out as possible with the squish pipette - I get about 450 μl of bottom phase (HNO_3) and 425 μl 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 ^{133}Cs.

☑ Transfer 0.5 ml of [*Stock*] to [5]

☑ Transfer 0.5 ml of [*Stock*] to [6]

☑ Transfer 0.5 ml of [*Stock*] to [7]

☑ Add scoop of sodium nitrite to [5]

☑ Add scoop of sodium nitrite to [6]

☑ Add scoop of sodium nitrite to [7]

☑ Centrifuged [5], [6] and [7] to push all solution to botttom of vials

☑ Start count of [5] noticed bubbles in solution, might have to recount - left overnight

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

2 Counting for Process 1 Mistake experiment (Alpha)

☑ Took 20 μl out of 3 and put onto planchet chip (no dillution)

- Moved chip too early (before drying, ruined detector volume)
- Made another source with an additional 20 μl, 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]

☑ Analyzed Counts from [5], [6], and [7]

- Did not like how [6] didn't fit with others

☑ Started recount of [6]

☑ Start Excel Sheet for analysis and write program for quicker gamma analysis

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

1 Preparation for 3 Cycles

☑ Finished count $\boxed{6}$

☑ 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

☑ $\boxed{5}$, $\boxed{6}$, and $\boxed{7}$ already in antichamber

☑ Transfer vials with clear lids into glovebox, but leave the blue lids in the antichamber (lid transfer area)

☑ Dump $\boxed{Back\ Ex\ Solution}$ into aqeuous waste ($\sim 0.2\ \mu$l) (decays - will prepare a fresh batch)

2 Counting for Process 1 Mistake experiment (Alpha)

☑ Moved alpha sample to count on PIPS detector

- 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

☑ Add 500μl TBP to $\boxed{5}$, $\boxed{6}$, $\boxed{7}$

☑ Shake $\boxed{5}$ on Pulse mode for 15 minutes

☑ Shake $\boxed{6}$ on Pulse mode for 15 minutes

☑ Shake $\boxed{7}$ on Pulse mode for 15 minutes

☑ Create $\boxed{EXBuddy}$ so all samples can be centrifuged together

- 500 μl of 4 M HNO_3 + 500 μl 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 $\boxed{5}$ aqueous transfer
  2. Sample $\boxed{6}$ organic transfer
  3. Sample $\boxed{7}$ 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 μl and transfered 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 μl) and transfered to $\boxed{5\ Aq}$, $\boxed{6\ Aq}$, and $\boxed{7\ Aq}$.

☑ Measure Volumes of 9 vials (Aqueous, organic, and original - units of μl)

- Clean outside of vials before taking volume measurements
- Centrifuge vials before taking volume measurements
- Google says that 1 drop of water is about 50 μl

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

☑ Count $\boxed{7\ Or}$ 12:00 pm - 6:00 pm

☑ Start count $\boxed{7\ Or}$ on face of detector 6:00 pm this is because I cannot see ^{134}Cs - the isotope I am most concerned about

- 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:

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

☑ 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

☑ Count $\boxed{6\ Or}$ 8:00 pm - 11:00 am

2 Contamination spill 10/25/16

☐ ~~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

☑ Clean up contamination from exploded vials in antichamber

- 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 $\sim$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 $\sim$ 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 - $\sim$100
- Glass vial - none

3 Cycle experiment, replicate of 3

☑ Count $\boxed{5\ Or}$ 3:00 pm - 7:30 pm (finally!!)

☐ ~~Count $\boxed{7\ Aq}$ 9:00 pm - 11:00 pm~~ (Spilled)

☑ Count $\boxed{6\ Aq}$ 7:00 pm - 9:00 pm

☑ Count $\boxed{5\ Aq}$ 9:00 pm - 8:00 am

☑ -

0.0417+/-0.0018 ml of 2.302+/-0.009 M Fe(II) in 0.0+/-0 M HNO_3 $\boxed{Stock\ Fe(II)}$
+
3.941+/-0.027 ml of 0.0+/-0 M Fe(II) in 4.06+/-0.05 M HNO_3 solution $\boxed{Fe\ Prep}$
+
4.000+/-0.020 ml of 0.0240+/-0.0010 M Fe(II) in 4.00+/-0.05 M HNO_3 solution
$\boxed{\rightarrow Bk\ Ex\ Solution}$.

☑ Add 430 μl Fe(II) solution to $\boxed{5\ Or}$

☑ Add 430 μl Fe(II) solution to $\boxed{6\ Or}$

☐ ~~Add XX μl Fe(II) solution to $\boxed{7\ Or}$~~ (spilled)

☑ Shake $\boxed{5\ Or}$ 15 minutes on pulse mode

☑ Shake $\boxed{6\ Or}$ 15 minutes on pulse mode

☐ ~~Shake $\boxed{7\ Or}$ 15 minutes on pulse mode~~ (spilled)

☐ ~~Remove XX μl organic and XX μl aqueous from $\boxed{Ex\ Buddy}$~~ (No longer necessary)

☑ Centrifuge $\boxed{5\ Or}$, $\boxed{6\ Or}$, ~~$\boxed{7\ Or}$~~ ~~$\boxed{Ex\ Buddy}$~~, 3,000 rpm for 5 minutes

☑ Vials labeled $\boxed{5\ AqII}$, $\boxed{5\ OrII}$, $\boxed{6\ AqII}$, $\boxed{6\ OrII}$, ~~$\boxed{7\ AqII}$~~, ~~$\boxed{7\ OrII}$~~, transfered into glovebox

☑ Separate phases for samples

- A total of 1 drops were dropped in this process
  1. Sample $\boxed{5\ Or}$ 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 transfered to $\boxed{5\ OrII}$, $\boxed{6\ OrII}$, and $\boxed{\cancel{7\ OrII}}$.
- Then as much of the top phase (organic) was removed as possible and transfered to $\boxed{5\ AqII}$, $\boxed{6\ AqII}$, and $\boxed{\cancel{7\ AqII}}$.

☑ Measure Volumes of 9 vials (Aqueous, organic, and original - units in μl)

Series	Aqueous II	Organic II	Original II	Should Add to	Missing
5	407+/-8.14	380+/-7.6	38+/-5	860+/-12.2	35.0+/-17.2
6	~~402~~415+/-8.3	~~360~~380+/-7.6	35+/-5	860+/-12.2	30+/-17.3

4 Calculation Work

☑ Updated Spreedsheets to calculate activities based on avaiable 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

☑ Finish count $\boxed{5\ Aq}$

☑ Start count $\boxed{6\ AqII}$

☑ Analyze current spectra

- Calculate activity (with error) for vials $\boxed{5}$, $\boxed{6}$, $\boxed{7}$, $\boxed{5\ A}$, $\boxed{5\ O}$, $\boxed{6\ A}$, $\boxed{6\ O}$, $\boxed{7\ O}$
- 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 ^{137}Cs ^{134}Cs 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

☑ Create graphic of all alpha spectra and locations of swipes

☑ 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}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}U: 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 ∼300 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
- ☐ 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

- ☑ Finish counting $\boxed{6\ AqII}$
- ☑ Start counting $\boxed{5\ AqII}$

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

☑ Finish count $\boxed{5\ AqII}$

☑ Checked math with John Burns

- The math was correct, but we noticed that Series 6 had larger D-values accros the board
- If we assume a 10 μl 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 ☑

☑ Start background count

☑ Go home, not feeling well

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"

- 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 ^{137}Cs...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

☑ This took a large portion of the day

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 → 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 ^{137}Cs, 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 ^{137}Cs

☑ Started a new background count

- It does look like he shielded ^{137}Cs

☑ 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 ^{137}Cs 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
- ☑ Assuming 10μl 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.
- ☐ Make Easy to read power point
- ☑ Automate Decay corrections

2 Details from research meeting

- Showed results, at first Chirayath, thought that ^{137}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

☑ Calculation for best volume to minimize error on D-values, several routes, averaged them

- The Weighted Mean

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

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

☐ Automate background calculation and decay corrections

2 Contamination spill 10/25/16

☑ Talked with Evgeny Tereshatov: ETereshatov@tamu.edu

- Said 52.50 ± 0.5 μCi decay corrected to 5 May, 2014 ^{144}Ce is to be disposed
- RSO 0079436
- Need Waste Disposal Report Form
- Made estimates on ^{137}Cs, ^{134}Cs
- Accidentally added ^{90}Sr, it should have been ^{125}Sb

☑ Called EHS three times, left message once - no response

Friday, 4 November 2016

1 Cycle experiment, replicate of 3

- ☑ 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 μl. (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!
- ☑ 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 ^{152}Eu count
- ☑ Start background (make sure things are okay)

Monday, 7 November 2016

1 Cycle experiment, round 2, replicate of 3

☑ Finish background count

☑ Practice transfer with 300 μl.

- A little frustrating
- Take a lunch break for headache, maybe second practice will go better
- Settled on 400 μl instead of 500 μl or 300 μl (happy medium)

☑ Create and label vials $\boxed{8}$, $\boxed{9}$, $\boxed{10}$, and $\boxed{Buddy}$, 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 ^{133}Cs. also, still using smaller vials, but will make sure to have double containment for transfer into glovebox

☑ Create $\boxed{Buddy}$ with ~~0.5~~ 0.4 (removed 0.1) ml of 4 M HNO_3 solution

☑ Put $\boxed{Buddy}$ inside a 15 ml vial, parafilm wrap

☑ -

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

☑ Parafilm wrap $\boxed{Stock\ Add}$

☑ Transfer $\boxed{Stock\ Add}$, $\boxed{8}$, $\boxed{9}$, $\boxed{10}$, $\boxed{Buddy}$, and $\boxed{closet}$ 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 HNO_3 solution $\boxed{closet}$
+
2.048+/-0.026 ml of 1.12+/-0.08 M HNO_3 solution $\boxed{Stock\ Add}$
=
2.500+/-0.025 ml of 4.00+/-0.05 M HNO_3 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. → *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 μl out of *Stock*, I would expect to get 690 μl out of *Stock*..where did 290 μl go? Did it evaporate? Do we need to parafilm wrap it?
- As a precaution, I will parafilm wrap it

☑ Transfer 400 μl *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

☑ Transfer *closet* to rad closet

☑ Transfer 0.4 ml of *Stock Add* to 8

☑ Transfer 0.4 ml of *Stock Add* to 9

☑ Transfer 0.4 ml of *Stock Add* to 10

☑ Add scoop of sodium nitrite to 8

☑ Add scoop of sodium nitrite to 9

☑ Add scoop of sodium nitrite to 10

☑ Put 8, 9, and 10 into 15 ml centrifuge tubes

☑ Centrifuged 8, 9 and 10 to push all solution to botttom of vials

☑ Fixed shielding on detector

- Retake background and efficiency count

☐ Note when ^{137}Cs 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

☑ Eff Count

☐ Practice extraction with 400 μl while doing counts tonight

☑ Count $\boxed{8}$

☑ Count $\boxed{9}$

☐ Count $\boxed{10}$

- Alarm didn't wake me up...didn't count $\boxed{10}$

Tuesday, 8 November 2016

1 Cycle experiment, round 2, replicate of 3

- ☑ Count $\boxed{10}$
- ☑ Label vials, $\boxed{8\ aq}$, $\boxed{8\ aq\ C}$, $\boxed{8\ or}$, $\boxed{8\ or\ C}$ $\boxed{9\ aq}$, $\boxed{9\ aq\ C}$, $\boxed{9\ or}$, $\boxed{9\ or\ C}$ $\boxed{10\ aq}$, $\boxed{10\ aq\ C}$, $\boxed{10\ or}$, $\boxed{10\ or\ C}$ (smaller 2.5 ml tubes)
- ☑ Label vials $\boxed{8\ mix}$, $\boxed{9\ mix}$, $\boxed{10\ mix}$ (smaller 1 ml tubes from John Burns, have conical bottoms, makes more minute separations easier)
- ☑ Transfer $\boxed{8}$, $\boxed{9}$, and $\boxed{10}$ into glovebox. With: $\boxed{8\ aq}$, $\boxed{8\ aq\ C}$, $\boxed{8\ or}$, $\boxed{8\ or\ C}$ $\boxed{9\ aq}$, $\boxed{9\ aq\ C}$, $\boxed{9\ or}$, $\boxed{9\ or\ C}$ $\boxed{10\ aq}$, $\boxed{10\ aq\ C}$, $\boxed{10\ or}$, $\boxed{10\ or\ C}$. (3 clear push caps, and 9 blue push caps). Also with 6 15 ml centrifuge tubes, and $\boxed{8\ mix}$, $\boxed{9\ mix}$, $\boxed{10\ mix}$
- ☑ Add 400 μl of $\boxed{TBP}$ to $\boxed{8}$, $\boxed{9}$, and $\boxed{10}$ each
- ☑ Vortex mix $\boxed{8}$ for 15 minutes on pulse mode
- ☑ Vortex mix $\boxed{9}$ for 15 minutes on pulse mode
- ☑ Vortex mix $\boxed{10}$ for 15 minutes on pulse mode
  - Switched to push caps for each of the above
- ☑ Centrifuge $\boxed{8}$, $\boxed{9}$, and $\boxed{10}$ with $\boxed{Buddy}$ on 3300 rpm, for 5 minutes
- ☑ 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 μl 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.

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

☐ Measure volumes of everything

☑ Transfer out $\boxed{8\ or\ C}$, $\boxed{8\ aq\ C}$, $\boxed{9\ or\ C}$, $\boxed{9\ aq\ C}$, $\boxed{10\ or\ C}$, $\boxed{10\ aq\ C}$, in 15 ml centrifuge tubes

☑ Radiac wash the above tubes, and store in fumehood behind lead - wait to count (Marianno has an experiment going on)

☑ Clean stuff in glovebox

☑ Start count $\boxed{10\ aq\ C}$ 4:00 pm

☑ Start count $\boxed{9\ aq\ C}$ 6:00 pm

☑ Start count $\boxed{8\ aq\ C}$ 8:00 pm

☑ Start count $\boxed{10\ or\ C}$ 10:00 pm - leave overnight

☑ Create graphic for experiment

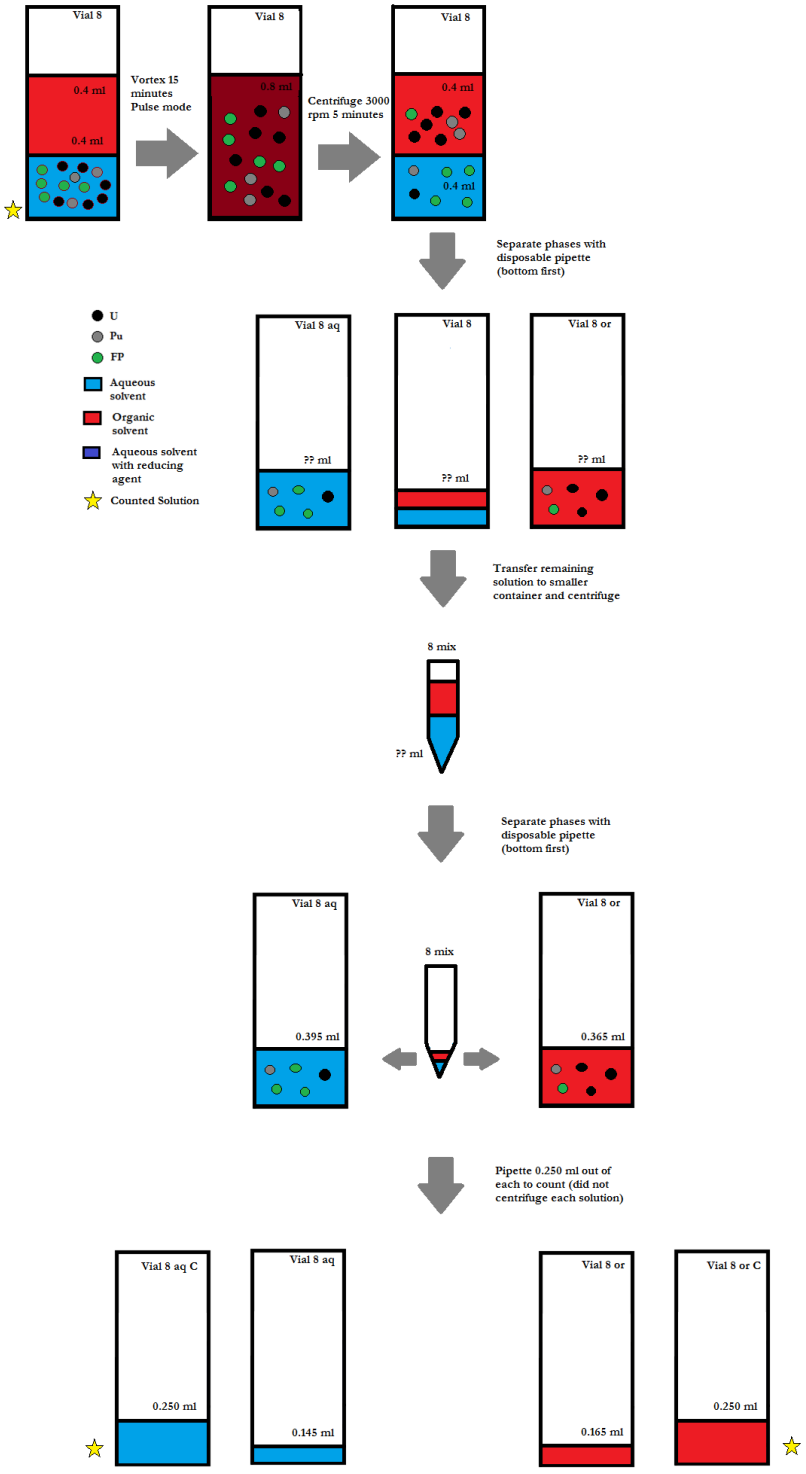

Figure 1: Extraction three times round 2 experimental setup

Wednesday, 9 November 2016

1 Cycle experiment, round 2, replicate of 3

☑ Finish count $\boxed{10 \text{ or } C}$

- Gave decent results for everything but ^{137}Cs

☑ Start count $\boxed{10 \text{ or } C}$ on face of detector

☑ Finish count $\boxed{10 \text{ or } C}$

☑ Start count $\boxed{9 \text{ or } C}$ on face of detector

☑ 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 ^{137}Cs
- Fix ^{154}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 $\boxed{9 \textit{ or } C}$
- ☑ Start count $\boxed{8 \textit{ or } C}$ on face of detector ($\sim$ 4 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 ^{134}Cs 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
- ☐ Respond to McClarren email - did this weekend
- ☐ Review McClarrens notes email - did this weekend
- ☐ Learn how to use ORIGEN - did this weekend

Friday, 11 November 2016

1 Cycle experiment, round 2, replicate of 3

☑ Finish count $\boxed{8 \textit{ or } C}$

☑ Start Efficiency count on face of detector

2 Things to do for school

☐ Find variances

☑ Learn how to use ORIGEN, and run it

☑ Learn how to change the things in ORIGEN

☐ 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

- The ^{137}Cs results look very small 10^{-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

☑ Counted $\boxed{8\ aq\ C}$, $\boxed{9\ aq\ C}$, and $\boxed{10\ aq\ C}$ on the face of the detector (only needed to count each like 10 minutes or so (40% dead time)

- To check and see if geometry is the issue (although these high dead times would probably give incorrect results - as Dr. Burns pointed out)

☑ Analyzed results from above counts, they increased the D-values to something more reasonable (similiar to the first experiment), but ^{137}Cs is still acting like a punk ($\sim 10^{-5}$)

- 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

☑ Recreate organic samples of $\boxed{8\ or\ C}$, $\boxed{9\ or\ C}$, and $\boxed{10\ or\ C}$. and count $\boxed{10\ or\ C}$ overnight

- 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 - $\boxed{8\ or}$ as opposed to $\boxed{8\ or\ C}$)
- Centrifuged both C and non-C containers for 5 minutes on highest setting (33)

- Repipetted out 250 μl 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

$$\begin{aligned}\frac{0.0129 \text{ g } DUO_2 \cdot 0.88 \cdot \frac{1}{238}}{0.005 \text{ Liters}} &= 0.009539M \\ 0.009539 \text{ M} \cdot \frac{0.5\ ml}{2.5\ ml} & \\ = 0.001908 \text{ M U} &\end{aligned}$$

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

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

Table 1: Values from Paper, 1.4 M U (much higher than ours 2 mM), and 3 M HNO_3 (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)

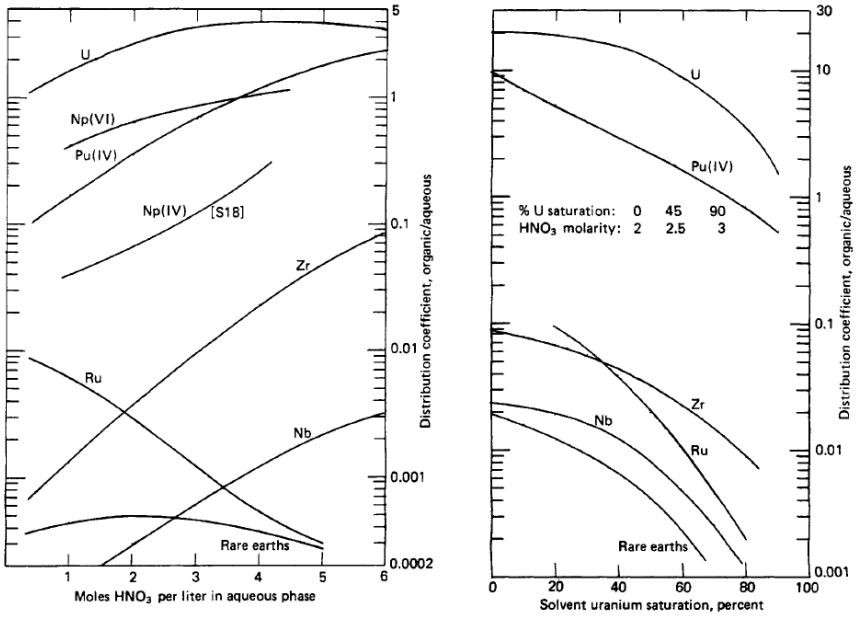

Figure 1: D value plots from Reactor handbook

- 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

☑ Looking at geometric differences between calibration source at 0 cm and 26 cm. and also between 10 *or* at 0 cm and 26 cm.

- Noticed there is a trend, might be able to use
- Also noticed that I counted my ^{152}Eu 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

☑ Stop count of $\boxed{10\ or\ C}$ around 5:50 am

☑ Start efficency count at 26 cm around 5:50 am

☑ Analyze results from $\boxed{10\ or\ C}$

- 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 $\boxed{10\ or\ C}$ and the second...WHY!?
- Maybe because some aqueous was in the original sample $\boxed{10\ or}$, 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 (also Mass Spec preparation

☑ Label vials: $\boxed{8, 9, 10\ Dillution}$, $\boxed{8\ aq\ Dillution}$, $\boxed{9\ aq\ Dillution}$, $\boxed{10\ aq\ Dillution}$, $\boxed{8\ or\ Dillution}$, $\boxed{9\ or\ Dillution}$, $\boxed{10\ or\ Dillution}$. Label Chips: $\boxed{8\ Chip}$, $\boxed{9\ Chip}$, $\boxed{10\ Chip}$, $\boxed{8\ aq\ Chip}$, $\boxed{9\ aq\ Chip}$, $\boxed{10\ aq\ Chip}$, $\boxed{8\ or\ Chip}$, $\boxed{8\ or\ Chip}$, $\boxed{10\ or\ Chip}$

☑ Also transfer 3 red and 4 blue push caps for smaller vials

☑ Transfer all above vials and chips into glovebox

3 Cycle experiment, round 2, replicate of 3

☑ Finish Eff count

- Rework calculations with new eff...didn't help much

☑ Start $\boxed{9\ or}$ count

☑ 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
- 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 $\boxed{8 \textit{ or } C}$ and $\boxed{9 \textit{ or } C}$

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
- ☑ take out the trash in the glovebox

1 Cycle experiment, round 2, replicate of 3

- ☑ Finish count $\boxed{9\ or}$
- ☑ Begin count $\boxed{8\ or}$ (9:44 am)

2 Cycle experiment, round 2, replicate of 3, ALPHA preparation (also Mass Spec preparation

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

$$\begin{gathered} 10\text{+/-}0.12\ \mu\text{l of } \boxed{Stock\ Add}\ (4\text{ M } HNO_3)\ \text{[smaller pipette]} \\ + \\ 990\text{+/-}9.9\ \mu\text{l of DI water (leftover in glovebox)} \\ = \\ 1\text{+/-}9.9\text{ ml of} \sim 0\text{ M } HNO_3\ \boxed{8, 9, 10\ Dillution} \end{gathered}$$

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

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

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

- ☑ Make alpha sample of each aqueous
  - ☑ - $\boxed{8\ aq}$

10+/-0.12 μl of $\boxed{8\ aq}$ (4 M HNO_3) [smaller pipette]
+
390+/-7.8 μl of DI water (leftover in glovebox)
=
0.4+/-0.0078 ml of $\sim$ 0 M HNO_3 $\boxed{8\ aq\ Dillution}$

- $\boxed{8\ aq}$ transfer contaminated gloves (had the blue push cap) and the vial accidentally fell

20+/-0.2 μl of $\boxed{8\ aq\ Dillution}$ dropped onto $\boxed{8\ aq\ Chip}$

☑ - $\boxed{9\ aq}$

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

10+/-0.12 μl of $\boxed{9\ aq}$ (4 M HNO_3) [smaller pipette]
+
390+/-7.8 μl of DI water (leftover in glovebox)
=
0.4+/-0.0078 ml of $\sim$ 0 M HNO_3 $\boxed{9\ aq\ Dillution}$

20+/-0.2 μl of $\boxed{9\ aq\ Dillution}$ dropped onto $\boxed{9\ aq\ Chip}$

☑ - $\boxed{10\ aq}$

10+/-0.12 μl of $\boxed{10\ aq}$ (4 M HNO_3) [smaller pipette]
+
390+/-7.8 μl of DI water (leftover in glovebox)
=
0.4+/-0.0078 ml of $\sim$ 0 M HNO_3 $\boxed{10\ aq\ Dillution}$

20+/-0.2 μl of $\boxed{10\ aq\ Dillution}$ dropped onto $\boxed{10\ aq\ Chip}$

☑ Make alpha sample of each organic phase

☑ - $\boxed{8\ or}$

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

20+/-0.2 μl of $\boxed{8\ or\ Dillution}$ dropped onto $\boxed{8\ or\ Chip}$

- **Spilled some organic on inner ring?? of** $\boxed{8\ or\ Chip}$**, question because hard to see in glovebox**

☑ - $\boxed{9\ or}$

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

10+/-0.12 μl of $\boxed{9\ or\ Dillution}$ dropped onto $\boxed{9\ or\ Chip}$

- **Changed volume on chip because** $\boxed{8\ or\ Chip}$ **potentially spilled over the inner ring**

☑ - $\boxed{10\ or}$

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

10+/-0.12 μl of $\boxed{10\ or\ Dillution}$ dropped onto $\boxed{10\ or\ Chip}$

- **Changed volume on chip because** $\boxed{8\ or\ Chip}$ **potentially spilled over the inner ring**

☑ **Note: Centrifuged all dillution vials before making alpha samples, which means that first all dillutions 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)

☑ Combine all aqueous phases together (done with disposable pipetets)

☑ $\boxed{8\ aq\ C}$ + $\boxed{8\ mix}$ → $\boxed{8\ aq}$ (take all of first and add to second)

☑ $\boxed{9\ aq\ C}$ + $\boxed{9\ mix}$ → $\boxed{9\ aq}$

☑ $\boxed{10\ aq\ C}$ + $\boxed{10\ mix}$ → $\boxed{10\ aq}$

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 ^{137}Cs values, looked at the first experiment, the one where I messed up, and liked the 110 value, now I am getting 10^{-5}...why?
- Dr. Burns suggested to increase the volume of the extraction phase (organic) to pin down the ^{137}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)

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

- 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

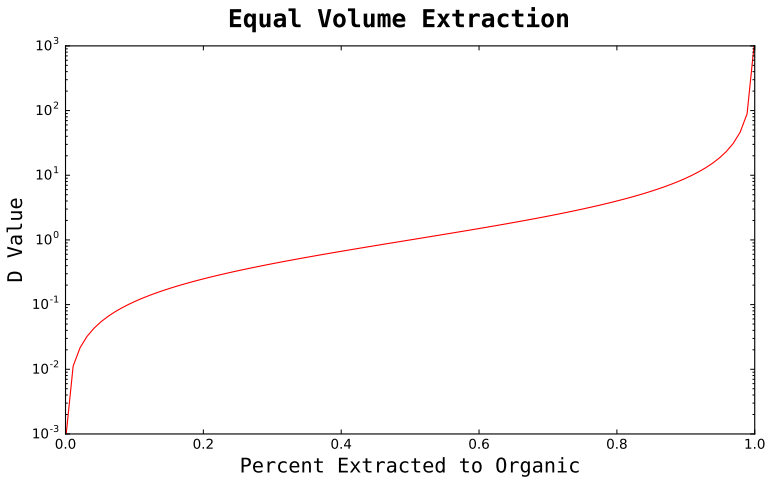

Figure 1: Percent extraction versus D value on log scale

Thursday, 17 November 2016

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

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

- ☑ Start Count $\boxed{10\ or\ Chip}$ (10:52 am)
- ☑ End Count $\boxed{10\ or\ Chip}$ Run time 7.54 hrs
- ☑ Start count $\boxed{10\ aq\ Chip}$ (6:29 pm)

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

In order to capture the D-value for ^{137}Cs, an experiment was proposed. Our problem with measuring ^{137}Cs 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 ^{137}Cs 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 μl, 4.47 μCi, $\sim$ 3.6% dead time
- $\boxed{6\ aq}$ has 469 μl, 4.40 μCi, $\sim$ 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 $\boxed{Stock}$, I know this because the activity density changed from $\boxed{Stock}$ and $\boxed{Stock\ add}$.
- If we take 800 μl total (after mixing $\boxed{5\ aq}$ and $\boxed{6\ aq}$), then we could expect $\sim$ 8.87 μl (about 200 cps), of ^{137}Cs 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)

$$\begin{aligned}\% =& \frac{1}{1+\frac{V_a}{V_o}\frac{1}{D}} \\ =& \frac{1}{1+\frac{1}{2}\frac{1}{0.01}} = 0.019\end{aligned}$$

This means if we double the volume of the organic, then we should get a decent count rate so as to count ^{137}Cs 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,

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

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

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

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

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

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.

- ☑ Combine $\boxed{5\ aq}$ and $\boxed{6\ aq}$ into $\boxed{5\ aq}$
- ☑ Take 800 μl out of $\boxed{5\ aq}$ and transfer into a 15 ml vial labeded $\boxed{56}$ (for some reason it was really difficult to get a precise volume - had to do many times)
- ☑ Start count $\boxed{56}$ at 26 cm

3 Cycle experiment, round 2, replicate of 3

☑ Finish count $\boxed{8\ or}$ ($\sim$ 9:45 am) about this time another count was started - vial 56, described above

☑ Analyzed last two organics, put into excel sheet

- All samples of organic, after mixing organic parts together, redrawing 250 μl and recounting, increased in activity. This could support the conclusion that some aqueous passed to the main organic, and when the 250 μl was first drawn, was on the bottom of the vial. When the 250 μl was second drawn, it had time to dissolve into the TBP, because HNO_3 is slightly soluble in TBP (Nuclear Chemical Engeineering pg 160)

4 Process Experiment (continuation from cycle experiment)

☑ Measure volumes of all aqueous phases, $\boxed{8\ aq}$ $\boxed{9\ aq}$, $\boxed{10\ aq}$

Table 1: 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...

☑ Label vials, $\boxed{8\ aqII}$, $\boxed{8\ aqII\ C}$, $\boxed{8\ orII}$, $\boxed{8\ orII\ C}$ $\boxed{9\ aqII}$, $\boxed{9\ aqII\ C}$, $\boxed{9\ orII}$, $\boxed{9\ orII\ C}$ $\boxed{10\ aqII}$, $\boxed{10\ aqII\ C}$, $\boxed{10\ orII}$, $\boxed{10\ orII\ C}$ (smaller 2.5 ml tubes)

- Will reuse $\boxed{8\ mix}$, $\boxed{9\ mix}$, $\boxed{10\ mix}$ (smaller 1 ml tubes from John Burns, have conical bottoms, makes more minute separations easier)

☑ Transfer: $\boxed{8\ aqII}$, $\boxed{8\ aqII\ C}$, $\boxed{8\ orII}$, $\boxed{8\ orII\ C}$ $\boxed{9\ aqII}$, $\boxed{9\ aqII\ C}$, $\boxed{9\ orII}$, $\boxed{9\ orII\ C}$ $\boxed{10\ aqII}$, $\boxed{10\ aqII\ C}$, $\boxed{10\ orII}$, $\boxed{10\ orII\ C}$. (3 clear push caps, and 6 blue push caps 6 red push caps). Also with 6 15 ml centrifuge tubes

☑ Add 397 μl of $\boxed{TBP}$ to $\boxed{8\ aq}$

☑ Add 389 μl of $\boxed{TBP}$ to $\boxed{9\ aq}$

☑ Add 396 μl of $\boxed{TBP}$ to $\boxed{10\ aq}$

☑ Vortex mix $\boxed{8\ aq}$ for 15 minutes on pulse mode

☑ Centrifuge $\boxed{8\ aq}$ with $\boxed{Buddy}$ at 3,300 rpm for 10 minutes

- Decieded after this to wait, and centrifuge them all together

☑ Vortex mix $\boxed{9\ aq}$ for 15 minutes on pulse mode

☑ Vortex mix $\boxed{10\ aq}$ for 15 minutes on pulse mode

☑ Centrifuge $\boxed{8}$, $\boxed{9}$, and $\boxed{10}$ 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 μl of each of the solutions A picture will be provided for the whole process for $\boxed{8\ aq}$ on the following page , below are specific notes about what occured during the experiment.

- $\boxed{8\ aq}$ was 248 μl pipetted to $\boxed{8\ aqII\ C}$ instead of 250 μl?

☐ ~~Measure volumes of everything~~

☑ Transfer out $\boxed{8\ orII\ C}$, $\boxed{8\ aqII\ C}$, $\boxed{9\ orII\ C}$, $\boxed{9\ aqII\ C}$, $\boxed{10\ orII\ C}$, $\boxed{10\ aqII\ C}$, in 15 ml centrifuge tubes

☑ Radiac wash the above tubes, and store in fumehood behind lead - wait to count

☑ Clean stuff in glovebox

☑ Start count $\boxed{9\ orII\ C}$ at 0 cm 4:06 pm

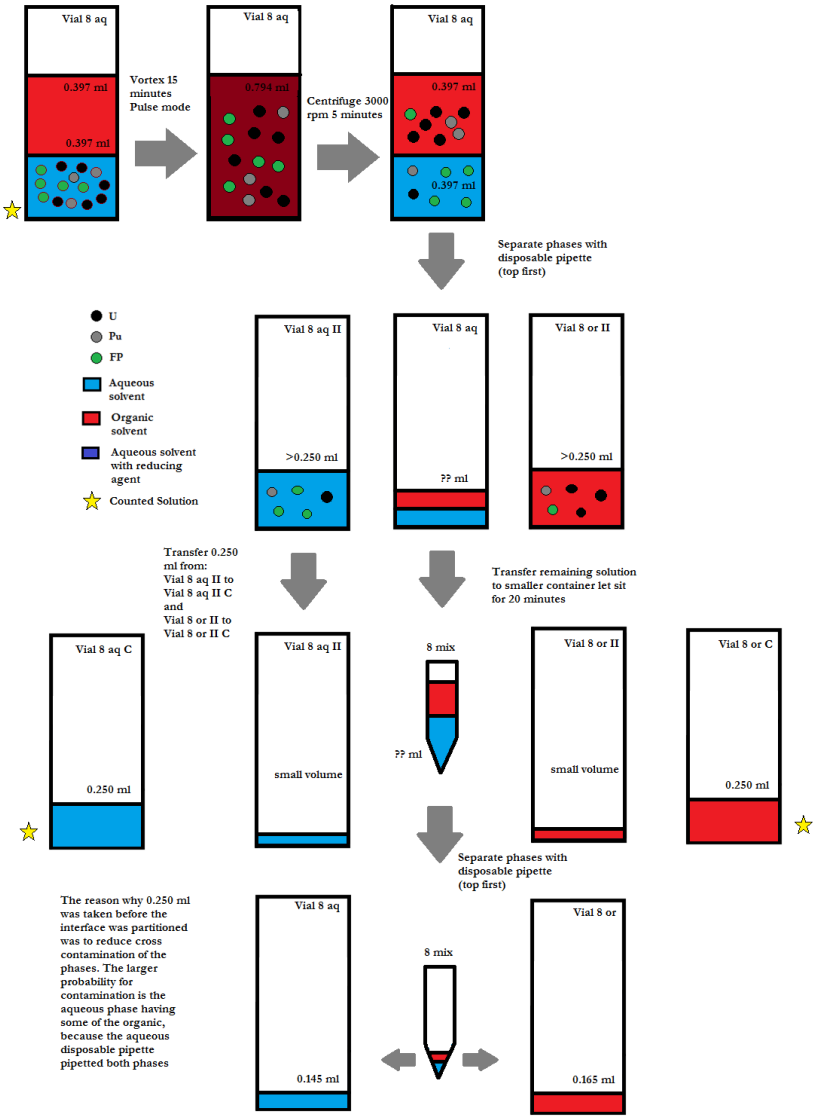

Figure 1: Extraction three times round 2 extraction 2

Friday, 18 November 2016

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

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

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

2 Process Experiment (continuation from cycle experiment)

- ☑ End count $\boxed{9\ orII\ C}$ RunTime 16.5 hr
- ☑ Start count $\boxed{8\ orII\ C}$ at 0 cm 8:56 am end around 11:15 pm (count $\boxed{56\ Big}$)
- ☑ End count $\boxed{8\ orII\ C}$ (RunTime 2.254 hrs)
- ☑ Start count $\boxed{10\ orII\ C}$ at 0 cm 1:04 pm

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

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

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

- ☑ Start count $\boxed{56\ Big}$ (11:17 am) to around 1:04 (started count $\boxed{10\ orII\ C}$

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

- ☑ Transfer $\boxed{56\ Big}$ into glovebox (wrapped in a ziplock bag so that less evaporation)
- ☑ Transfer 800 μl out of $\boxed{56\ Big}$ into $\boxed{56}$
- ☑ Add 8.0 μl of TBP to $\boxed{56}$
- ☑ Shake $\boxed{56}$ on vortex mixer for 15 minutes
- ☑ Convert a 2.5 ml vial holder (a 15 ml tube) to a buddy, by adding 800 μl of DI water and 8.0 ml of TBP to it...scratch out label. $\boxed{56\ Buddy}$
- ☑ Centrifuge $\boxed{56}$ with $\boxed{56\ Buddy}$ for 15 minutes at 3,300 rpm
- ☑ Carefully pipette with disposable pipette, as much of top phase of $\boxed{56}$ as possible to $\boxed{56\ Or}$ (50 ml tube)
- ☑ Carefully pipette with new disposable pipette, the bottom phase of $\boxed{56}$ (aq) to $\boxed{56\ aq}$ (15 ml tube)
- ☑ Clean up work area in glovebox
- ☑ Transfer interface of $\boxed{56}$ to $\boxed{56\ mix}$, seal and let sit for the time being
- ☑ Transfer out of glovebox $\boxed{56\ aq}$ and $\boxed{56\ or}$. Clean with radiac wipes
- ☑ Count $\boxed{56\ or}$ (2:28 pm), expecting 20 cps (calculation below, where 0.01 is the expected D-value of ^{137}Cs)

$$\frac{200\ \text{cps}_{\text{aq}}}{800\ \mu\text{l}} \cdot 8,000\ \mu\text{l} \cdot 0.01 = 20\ \text{cps}_{\text{or}}$$

Sadly, first glance gives around 0.1 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 (also Mass Spec preparation

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

☑ End Count $\boxed{9\ aq\ Chip}$ Run time 18.5 hrs

☑ Start Count $\boxed{10\ Chip}$ (1:34 pm)

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

☑ End count $\boxed{56\ or}$

☑ Start count $\boxed{56\ aq}$ (1:34 pm)

Monday, 21 November 2016

- ☑ Update laboratory notebook with all the experiments from last week, took most of the morning
- Dr. Mariannos experiment today started around 3:00 pm

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

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

- ☑ End Count $\boxed{10\ Chip}$ Run time 18.5 hrs
- ☑ Start Count $\boxed{9\ Chip}$ (10:00 am)
- ☑ End Count $\boxed{9\ Chip}$ Runtime 5.3 hrs
- ☑ Start Count $\boxed{8\ aq\ Chip}$ (3:21 pm)

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.

- ☑ Start count $\boxed{9\ aqII}$ (10:02 am)
- ☑ End count $\boxed{9\ aqII}$ Runtime 5.02 hr
- ☑ Start count $\boxed{9\ aqII}$ (3:25 pm) - yes I accidentally counted the same thing twice, there is a lot going on

Tuesday, 22 November 2016

- ☐ Modify spreadsheet so that the three errors can be minimized
- ☐ Find references for D-values

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

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

- ☑ End Count $\boxed{8\ aq\ Chip}$ Run time 17.1 hrs
- ☑ Start Count $\boxed{8\ Chip}$
- ☑ End Count $\boxed{8\ Chip}$
- ☑ Start Count $\boxed{8\ or\ Chip}$

2 Process Experiment (continuation from cycle experiment)

- ☑ End count $\boxed{9\ aqII\ C}$ Runtime 16.7 hr
- ☑ Start count $\boxed{8\ aqII\ C}$
- ☑ End count $\boxed{8\ aqII\ C}$
  - Start Count from other experiment

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

- ☐ Start with analysis of first extraction
  - Things are discouraging...still getting D of 10^{-5} for calculation using organic and calculation using aqueous
  - The other elements are within reason

- **What is different between my experiments and Jarrod's past experiments?**
- Also note, 15 ml tube has about 3% difference from 50 ml tube

☑ Create new TBP, 15 ml TBP + 35 ml kerosene → $\boxed{TBP\ Remake}$

☑ Transfer $\boxed{TBP\ Remake}$ and $\boxed{56\ aq}$ into glovebox

☑ Measured volume of $\boxed{56\ aq}$ to be 700 μl...why it so low? We had some evaporation?

☑ Add 7.0 ml of $\boxed{TBP\ Remake}$ to $\boxed{56\ aq}$

☑ Shake $\boxed{56\ aq}$ for 15 minutes on pulse mode

☑ Create vials $\boxed{56\ AqII}$ (15 ml) and $\boxed{56\ OrII}$ (50 ml) and $\boxed{56\ mixII}$ and transfer into glovebox

☑ Create a $\boxed{Buddy}$ for centrifuging (unlabled - sorry!)

☑ Centrifuge $\boxed{56\ aq}$ with $\boxed{Buddy}$ for 15 minutes at 3,300 rpm

☑ Separate (top phase first) $\boxed{56\ aq}$ into $\boxed{56\ AqII}$, $\boxed{56\ OrII}$, and $\boxed{56\ mixII}$

☑ Transfer $\boxed{56\ AqII}$ and $\boxed{56\ OrII}$ out of glovebox, clean with radiac wipes

☑ Start counting $\boxed{56\ OrII}$ at 26 cm away from detector

- Initially looks like the sample still has 10^{-5} for ^{137}Cs

Wednesday, 23 November 2016

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

☑ Stop count $\boxed{56\ orII}$

☐ Analyze data from 56 experiment

2 Process Experiment (continuation from cycle experiment)

☑ Start Count $\boxed{10\ aqII\ C}$

☐ Analyze second extraction data

3 Cycle experiment, round 2, replicate of 3, ALPHA preparation (also Mass Spec preparation

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

☐ End Count $\boxed{8\ or\ Chip}$

- Accidentally cleared data...stupid, recounting

☑ Start count $\boxed{8\ or\ Chip}$ again

☐ 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.

Break 24-27, November 2016

1 Process Experiment (continuation from cycle experiment)

☑ Stop Count $\boxed{10\ aqII\ C}$

☑ Start Count $\boxed{8\ aqII\ C}$

2 Cycle experiment, round 2, replicate of 3, ALPHA preparation (also Mass Spec preparation

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

☑ Stop count $\boxed{8\ or\ Chip}$ SAVE! shut down alpha 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

Monday 28, November 2016

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

- ☐ Analyze data from 56 experiment
- ☐ Review Dr. Chirayath's paper
- ☐ Review my old paper and see why its going wonky

2 Process Experiment (continuation from cycle experiment)

- ☑ Stop count $\boxed{8\ aqII\ C}$
- ☑ Start background count
- ☐ Analyze second extraction data

3 Cycle experiment, round 2, replicate of 3, ALPHA preparation (also Mass Spec preparation

- ☐ Analyze Data from alpha spectrum