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
- 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 → $\boxed{Stock\ HNO_3}$
  - 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 $Fe(NH_2SO_3)_2$ → $\boxed{Stock\ Fe(II)}$
  - 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)

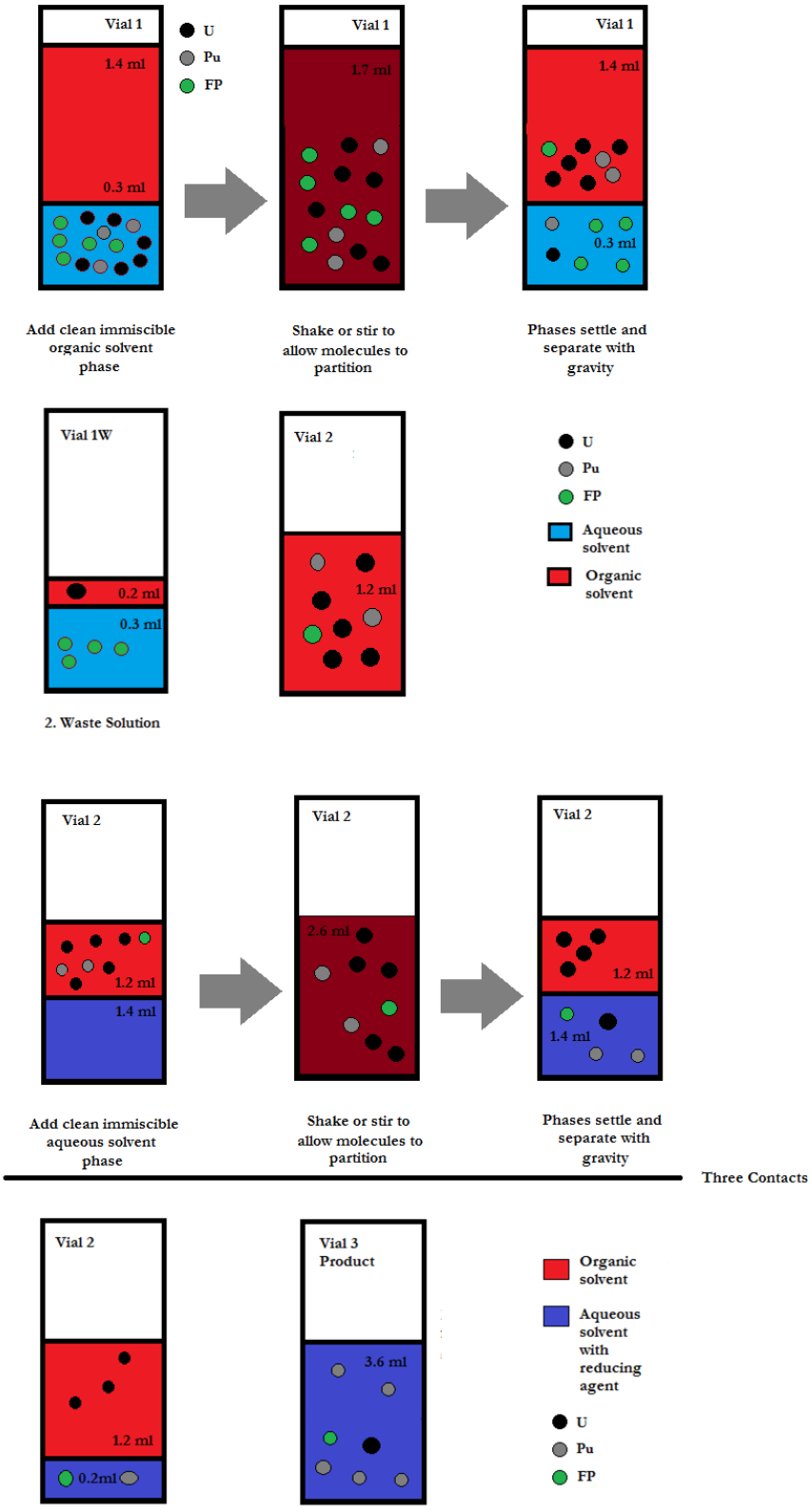

Figure 1: Process 1 Mess Up Experimental Overview

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

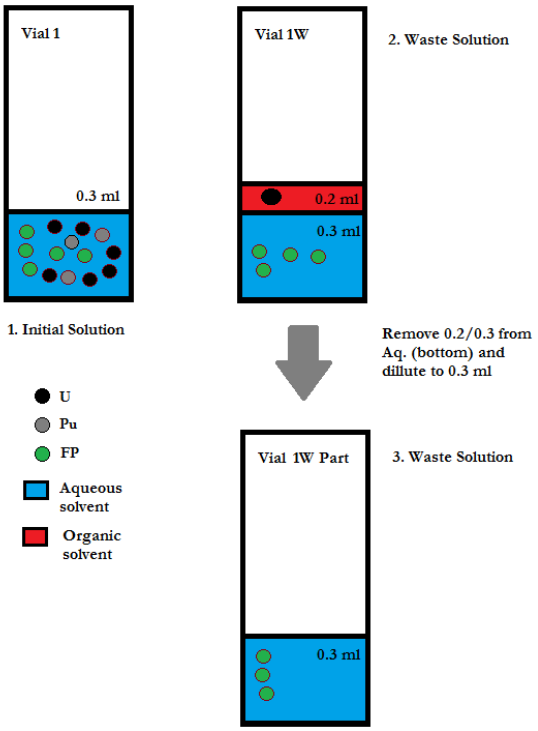

Figure 1: First Three Counts

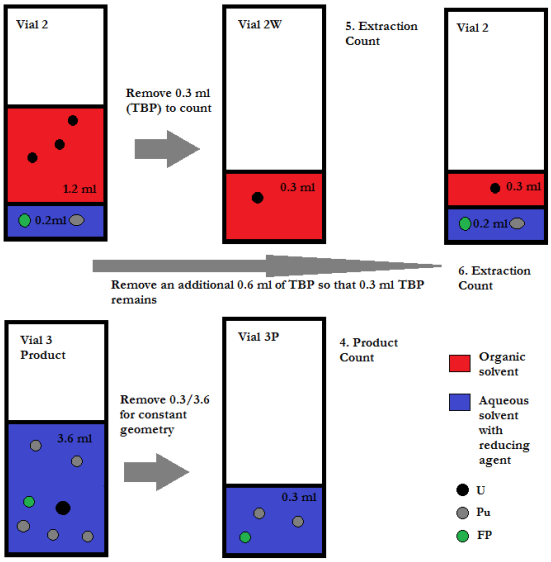

Figure 2: Second Three Counts

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)
=
0.2 ml of $\sim$ 0 M HNO_3 $\boxed{4\ \textit{Dillution}}$

☑ Prepare and count alpha sample of Stock

- Take 20 μl of $\boxed{4\ \textit{Dillution}}$, 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 30,000 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

☐ ~~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 - ∼100
- Glass vial - none

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

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

☐ Count $\boxed{6\ Aq}$ 11:00 pm - 1:00 am

☐ Count $\boxed{5\ Aq}$ 1:00 am - 3:00 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 XX μl Fe(II) solution to $\boxed{5\ Or}$

☐ Add XX 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 $\boxed{5\ Or}$, $\boxed{6\ Or}$, ~~$\boxed{7\ Or}$~~ ~~$\boxed{Ex\ Buddy}$~~, 30,000 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 XX drops were dropped in this process
  1. Sample $\boxed{XX}$ 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 $\boxed{5\ OrII}$, $\boxed{6\ OrII}$, and ~~$\boxed{7\ OrII}$~~.

- Then as much of the bottom phase (aqueous) was removed as possible (turns out to be around 430 μl) 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	Add to
5				
6				

☐ Count Vials

☐ Analyze spectra

2 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.