## **Laboratory Journal**

## Paul Mendoza

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This notebook begins 6 October 2016

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

#### 1 Isotopes we are looking for

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

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

Table 1: Isotope solve list.

### 2 Experiment Notes

- Project Number: 504370-0001
- Files on computer saved in C:/Paul\_Mendoza

- <sup>152</sup>Eu Liquid calibration source
  - Source 1577-22
  - 497.0 nCi
  - Assy Date: 15 Feb 12
  - -1.00568g
- Stock HNO<sub>3</sub>: 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+/-0.5Molar 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  $1000\mu$ l pipett from glovebox
- Decontaminate with radic dump waste into glass aq rad outside glove box
- Practice pipetting  $500\mu$ l to glass vial setting  $503 \mu$ l gives  $500 \mu$ l
- Class/lunch Break
- Get alpha detector from Dr. Marianno
- Set up laboratory notebook

• Calculation To do calculation to determine the volumes needed for a final concentration of a particular volume, knowing the initial concentrations

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

Where:

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

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

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

$$b_2 = M_3V_3$$

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

$$B = (1 - wt\%_3)V_3\rho_3 - (1 - wt\%_1)V_1\rho_1$$

$$A = M_3V_3 - M_1V_1$$

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

Need iterative solution, choose:

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

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

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

#### 1 Stock creation

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

**√** \_

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

**✓** -

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

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

#### 2 Preparation for Process 1

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

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

✓ Finish background count, lasted 12 hours

#### 1 Preparation for Process 1

centrifuging

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

$$V_1$$
 ml of  $M_{1,Fe}$  Fe(II) in  $M_{1,HNO_3}$  HNO<sub>3</sub> +  $V_2$  ml of  $M_{2,Fe}$  Fe(II) in  $M_{2,HNO_3}$  HNO<sub>3</sub> =  $V_3$  ml of  $M_{3,Fe}$  Fe(II) in  $M_{3,HNO_3}$  HNO<sub>3</sub>.

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<sub>3</sub> constant:  $V_2=\frac{V_3M_{3,HNO_3}}{M_{2,HNO_3}}$   
Mass Constant:  $V_2=\frac{V_3\rho_3-V_1\rho_1}{\rho_2}$ 

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

Solve iteratively (where  $M_{2,HNO_3}$  determines  $\rho_2$ ) 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<sub>3</sub> solution,
- Transfer two smaller vials (one for TBP phase), one for Fe phase, with two different lids into glovebox (with a larger vial to hold them in the centrifuge)
- Transfer two smaller vials with centrifuge vials for centrifuging, keep one with water 0.3 ml, and TBP mix 0.32 ml  $\boxed{Vial~1~Budd}$ , and the second with 1.2 ml of TBP mix and 1.25 ml water  $\boxed{Vial~2~Budd}$
- Transfer Stock and  $\boxed{1}$  to glovebox
- Transfer another vial to hold the Fe solution
- Make sure tweezers are in glovebox (they are) to remove smaller vials from centrifuge tubes
- Transfer slightly contaminated pipette to glovebox
- All above vials that would contain solution were rinsed with whatever they would hold for approximately 3 minutes

**1** \_

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

**✓** \_

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

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

\_ -

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

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

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

#### 1 Process 1 Mistake experiment

✓ First contact - Extraction

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

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

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

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

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

4.000+/-0.020 ml of 0.0240+/-0.0010 M Fe(II) in 4.00+/-0.05 M HNO<sub>3</sub> solution  $\rightarrow Bk\ Ex\ Solution$ .

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

This experiment had sputtering of pipette at certain times.

### 2 Counting for Process 1 Mistake experiment (Gamma)



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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)





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)

- $\square$  Finish count  $\boxed{3P}$
- - Determined <sup>137</sup>Cs, <sup>144</sup>Ce, <sup>106</sup>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,  $\mu Ci$
- ✓ Notes for research meeting
  - Process dilutes by factor of 12, no matter what

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

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

#### 1 Counting for Process 1 Mistake experiment (Gamma)

✓ Finish count 2W

### 2 Counting for Process 1 Mistake experiment (Alpha)

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

- - From Jarrod's stock  $10\mu l$  was dilluted to 1ml and 10  $\mu l$  was taken

$$\begin{array}{c} 10~\mu l~of~ \boxed{Stock}~(4~M~HNO_3)\\ +\\ 190~\mu l~of~DI~water~(leftover~in~glovebox)\\ =\\ 0.2~ml~of\sim 0~M~HNO_3~ \boxed{4~Dillution} \end{array}$$

- - It should be noted that once an alpha source is placed on these disks and dried out, they look no different from other disks
- Let dry in glovebox

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

Finish count 2

### 2 Counting for Process 1 Mistake experiment (Alpha)

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

### 3 Analysis for Process 1 Mistake (Gamma)

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

## Monday - Wednesday, 17-19 October 2016

### 1 Analysis for Process 1 Mistake (Gamma)

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

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

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

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

$$CPS = A\gamma\epsilon$$

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

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

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

#### ✓ Notes for Research Meeting

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

## Thursday, 20 October 2016

#### 1 Preparation for 3 Cycles

Note from John:

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

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

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

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

## Friday, 21 October 2016 9:30am - 12:00 pm 1:00 pm 5:00 pm

✓ Updated this lab notebook (most of this morning)

### 1 Preparation for 3 Cycles

- $\hfill\Box$  Practice pipetting out with squish tops like John Mentioned
  - Determine if 0.3 ml is a good amount of solution to use