Laboratory Journal

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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 Cycle 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 Cycle 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 Cycle 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 Cycle 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 1.4 $\boxed{Bk\ Ex\ Solution}$ to $\boxed{2}$
- Shake pulse mode for 15 minutes
- Remove 1.2 ml of bottom phase (Fe(II)) $\boxed{\rightarrow 3}$

☑ Back Extraction - Third 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)) $\boxed{\rightarrow 3}$

This experiment had sputtering of pipette at certain times.

2 Counting for Cycle 1 Mistake experiment

☑ 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)

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

1 Counting for Cycle 1 Mistake experiment

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 $\boxed{4\ M\ HNO_3}$ $\boxed{\rightarrow 1W}$
  - 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 0.4 ml, 0.2 ml of TBP, and 0.2 ml of HNO_3

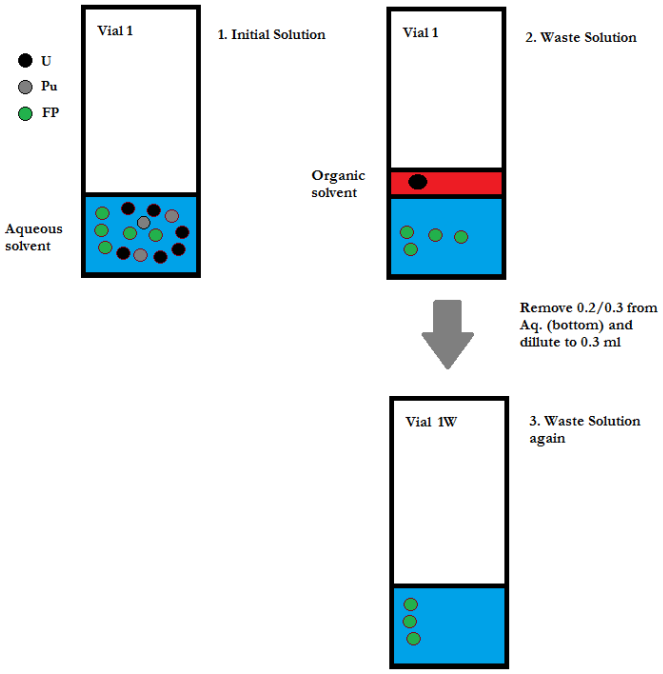

Figure 1: First Three Counts

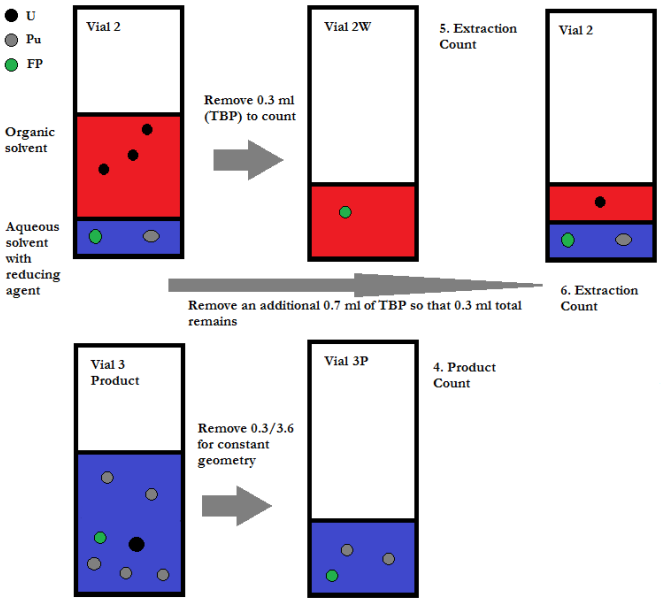

Figure 2: Second Three Counts

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

1 Counting for Cycle 1 Mistake experiment

☑ 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 Cycle 1 Mistake experiment

- ☑ Finish count $\boxed{2W}$
- ☑ Start count $\boxed{2}$
- ☑ Fix alpha counter, reivew alpha counting
  - Alpha detector broken, fixed by plugging into proper port
  - Counted Calibration Alpha source
    - item
  - Counted Background

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

1 Counting for Cycle 1 Mistake experiment

☑ Finish count 2