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# Laboratory Journal

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

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

Table 1: Isotope solve list.

## **2 Experiment Notes**

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

- Stock  $\text{HNO}_3$ : Assuming Temp=24.8+/-3  $\rightarrow$  *Stock HNO<sub>3</sub>*
  - 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  $\text{Fe}(\text{NH}_2\text{SO}_3)_2 \rightarrow$  *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 $\mu\text{l}$  pipett from glovebox
- Decontaminate with radic - dump waste into glass aq rad outside glove box
- Practice pipetting 500 $\mu\text{l}$  to glass vial - setting 503  $\mu\text{l}$  gives 500  $\mu\text{l}$
- 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:

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

$$V_2 = V_3 - V_1$$

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

✓ -

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

✓ -

$$\begin{aligned} &\text{Combine } 0.500 \pm 0.005 \text{ ml of } 15.43 \pm 0.06 \text{ M HNO}_3 \text{ solution } \boxed{\text{closet}} \\ &+ \\ &2.048 \pm 0.026 \text{ ml of } 1.12 \pm 0.08 \text{ M HNO}_3 \text{ solution } \boxed{\text{Stock}} \\ &= \\ &2.500 \pm 0.025 \text{ ml of } 4.00 \pm 0.05 \text{ M HNO}_3 \text{ solution. } \boxed{\rightarrow \text{Stock}} \end{aligned}$$

✓ Lock  $\boxed{\text{Stock}}$  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

Friday, 7 October 2016

9:00am - 12:00 am

1:00pm - 4:00pm

- ✓ 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 furthest 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 `Stock` transfer to `1` for counting
  - `1` is a smaller tube, which will fit into a larger centrifuge tube for, well, centrifuging
  - `1` tube cannot fit into centrifuge tube with white push cap (pushes on outside of tube), white push cap is necessary when vortex 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 `1` for 1 hour and 24 minutes
- ✓ Fix density calculation in code, was slightly wrong before, this means `Stock` and `1` are slightly different from what they should be, but within error
- ✓ Calculation for creation of Fe(II) solution (next page)



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

The knowns are:

$$M_{1,Fe} = 2.302, \rho_1 = 1.418, M_{1,HNO_3} = 0 \text{ (Fe Stock solution)}$$

$$M_{2,Fe} = 0, \rho_2 = \rho_{HNO_3}(M_{2,HNO_3})$$

$$V_3 = 4 \text{ ml}, M_{3,Fe} = 0.024, M_{3,HNO_3} = 4, \rho_3 = \rho_{HNO_3}(4M)$$

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

$$\text{Mols of HNO}_3 \text{ constant: } V_2 = \frac{V_3M_{3,HNO_3}}{M_{2,HNO_3}}$$

$$\text{Mass Constant: } V_2 = \frac{V_3\rho_3 - V_1\rho_1}{\rho_2}$$

$$\text{Combine last two equations: } M_{2,HNO_3} - \frac{V_3M_{3,HNO_3}\rho_2}{V_3\rho_3 - V_1\rho_1} = 0$$

$$\text{Solve iteratively (where } M_{2,HNO_3} \text{ determines } \rho_2) \text{ with first guess of: } M_{2,HNO_3} = \frac{M_{3,HNO_3}V_3}{V_2}$$

**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<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 Vial 1 Budd, and the second with 1.2 ml of TBP mix and 1.25 ml water Vial 2 Budd
- Transfer Stock and 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

✓ -

$$\begin{aligned} &15 \pm 0.15 \text{ ml of TBP } \boxed{\text{Stock TBP}} \\ &+ \\ &35 \pm 0.35 \text{ ml of kerosene } \boxed{\text{Stock kerosene}} \\ &= \\ &50 \pm 0.5 \text{ ml of 30 vol.\% TBP. } \boxed{\rightarrow \text{TBP}} \end{aligned}$$

✓ -

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

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  $HNO_3$ ).

☐ -

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

☒ 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 Cycle 1 experiment

✓ First contact - Extraction

- Add 0.32 ml  $TBP$  to  $1$
- 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  $TBP$  to  $1$  (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)  $\rightarrow 2$

✓ Creation of *Bk Ex Solution*

0.0417+/-0.0018 ml of 2.302+/-0.009 M Fe(II) in 0.0+/-0 M HNO<sub>3</sub> *Stock Fe(II)*  
 +  
 3.941+/-0.027 ml of 0.0+/-0 M Fe(II) in 4.06+/-0.05 M HNO<sub>3</sub> solution *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  
→ *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)) → 3
  - Lost two drops
  - While placing vial into centrifuge, cap shot off, spraying solution everywhere...great

✓ Back Extraction - Second Contact

- Add 1.4 *Bk Ex Solution* to 2
- Shake pulse mode for 15 minutes
- Remove 1.2 ml of bottom phase (Fe(II)) → 3

✓ Back Extraction - Third Contact

- Add 1.4 *Bk Ex Solution* to 2
- Shake pulse mode for 15 minutes
- Remove 1.2 ml of bottom phase (Fe(II)) → 3

This experiment had sputtering of pipette at certain times.

## 2 Counting for Cycle 1 experiment

✓ Counted waste 1, containing 0.3 ml of *Stock* and 0.2 ml *TBP*, on HPGe, for 12 hours (left for the night)

**Tuesday, 11 October 2016**  
**9:00 pm - 11:00 pm**

## **1 Counting for Cycle 1 experiment**

There are 6 things to count.

- ☒ Initial solution  $\boxed{1}$  - 23 cm away, 0.3 ml  $\text{HNO}_3$
- ☒ Waste  $\boxed{1}$  - 23 cm away, 0.3 ml  $\text{HNO}_3$  0.2 ml TBP
- ☐ Pull out 0.2 from bottom of  $\boxed{1}$  ( $\text{HNO}_3$ ), dilute to 0.3 ml with 4 M  $\text{HNO}_3$   $\boxed{\rightarrow 1W}$
- ☐ Pull out 0.3 ml from  $\boxed{3}$  to count  $\boxed{\rightarrow 3P}$  (product)
- ☐ 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  $\text{HNO}_3$