
Laboratory Journal

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Beginning 6 October 2016

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1 Experiment Notes

- Project Number: 504370-0001
- Files on computer saved in C:/Paul_Mendoza
- Stock HNO_3 : Assuming Temp= $24.8 \pm 3 \rightarrow \boxed{\text{StockHNO}_3}$
 - 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 $\text{Fe}(\text{NH}_2\text{SO}_3)_2 \rightarrow \boxed{\text{StockFe(II)}}$
 - 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

2 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 - 5:00pm

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

✓ -

$$\begin{aligned} &0.149 \pm 0.011 \text{ ml of } 15.43 \pm 0.06 \text{ M HNO}_3 \text{ } \boxed{StockHNO_3} \\ &+ \\ &1.91 \pm 0.08 \text{ ml of } 0.0 \pm 0 \text{ M solution } \boxed{DIWater} \\ &= \\ &2.048 \pm 0.026 \text{ ml of } 1.12 \pm 0.08 \text{ M HNO}_3 \text{ solution } \boxed{\rightarrow Stock} \text{ (glass container)} \end{aligned}$$

✓ -

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

✓ 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

Friday, 7 October 2016

9:00am - 12:00 am

1:00pm - 5:00pm

- ✓ 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 Cycle 1 experiment

- ✓ 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 Cycle 1 experiment

- ✓ 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

Sunday, 9 October 2016
7:00 pm

1 Cycle 1 experiment

- ☐ 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
 - Transfer two smaller vials with centrifuge vials for centrifuging, keep one with water 0.3 ml, and TBP mix 0.32 ml, and the second with 1.2 ml of TBP mix and 1.3 ml water
 - Transfer Stock and 1 to glovebox
 - Transfer another vial to hold the Fe solution
 - Make sure tweezers are transferred in as well - to remove smaller vials from centrifuge tubes

☐ -

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

☐ -

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

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).

Sunday, 9 October 2016

7:00 pm

□ -

0.0417+/-0.0018 ml of 2.302+/-0.009 M Fe(II) in 0.0+/-0 M HNO₃ *Stock Fe(II)*
+
3.941+/-0.027 ml of 0.0+/-0 M Fe(II) in 4.06+/-0.05 M HNO₃ solution *Fe Prep*
+
4.000+/-0.020 ml of 0.0240+/-0.0010 M Fe(II) in 4.00+/-0.05 M HNO₃ solution