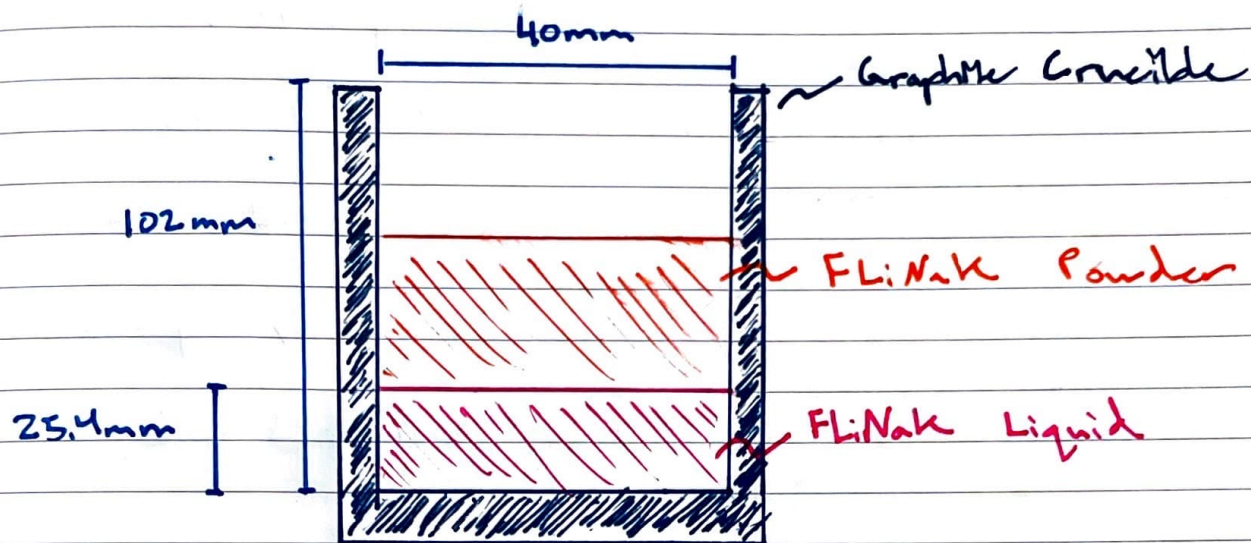


Day 67

10/4/22

- Ask Barrett what time is your operation
- Graphite FLiNak fill calculations



Volume Required for Liquid FLiNak:

$$V = \pi (20\text{mm})^2 (25.4\text{mm})$$

$$V = 31,918.58\text{mm}^3 = 3.191858 \times 10^{-5}\text{m}^3$$

Density of FLiNak at 650°C ~ 2,050 kg/m<sup>3</sup>

Amount of FLiNak powder required:

$$\frac{3.191858 \times 10^{-5}\text{m}^3}{1} \cdot \frac{2,050\text{kg}}{1\text{m}^3} = 0.065433089\text{kg}$$

65.43 g FLiNak

Day 67 (Continued)

10/4/22

- In order to make 65.43 g FLiNaK we need to measure out our constituents...

Eutectic FLiNaK has a molar mass of 41.291 g/mol

$$\frac{65.43 \text{ g FLiNaK}}{1} \cdot \frac{1 \text{ mol FLiNaK}}{41.291 \text{ g FLiNaK}} = 1.5846 \text{ mol FLiNaK}$$

For 1.5846 mol FLiNaK we need... (Eutectic)

$$1.5846 \text{ mol FLiNaK} \times 0.465 = 0.736839 \text{ mol LiF}$$

$$\frac{0.736839 \text{ mol LiF}}{1} \cdot \frac{25.939 \text{ g LiF}}{1 \text{ mol LiF}} = \boxed{19.11 \text{ g LiF}}$$

$$1.5846 \text{ mol FLiNaK} \times 0.115 = 0.18223015 \text{ mol NaF}$$

$$\frac{0.18223015 \text{ mol NaF}}{1} \cdot \frac{41.988 \text{ g NaF}}{1 \text{ mol NaF}} = \boxed{7.65 \text{ g NaF}}$$

$$1.5486 \text{ mol FLiNaK} \times 0.420 = 0.665532 \text{ mol KF}$$

$$\frac{0.665532 \text{ mol KF}}{1} \cdot \frac{58.096 \text{ g KF}}{1 \text{ mol KF}} = \boxed{38.66 \text{ g KF}}$$

To check my work...

$$19.11 \text{ g LiF} + 7.65 \text{ g NaF} + 38.66 \text{ g KF} = 65.42 \text{ g FLiNaK}$$

⇒

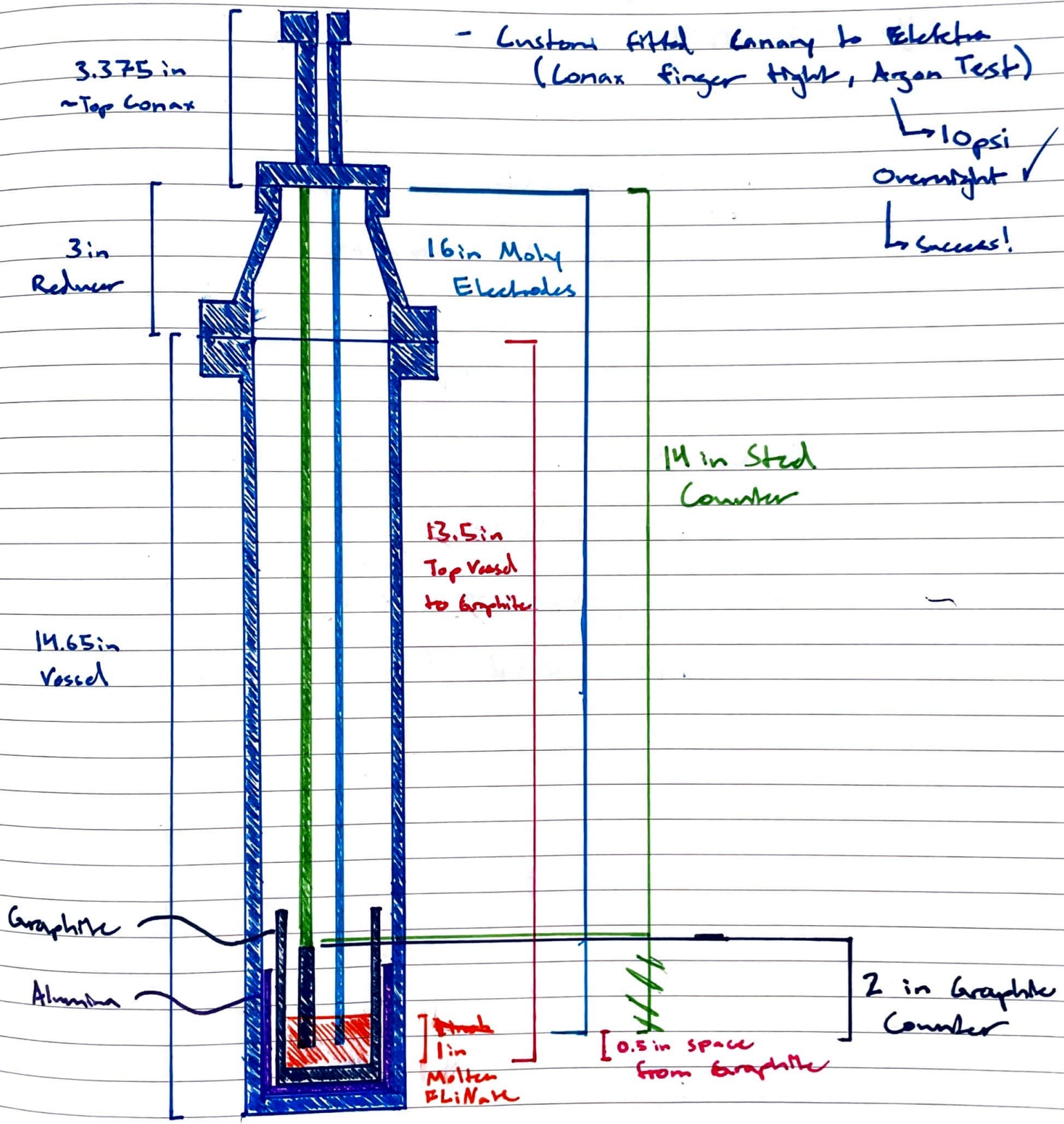
~~65.46~~

$$65.42 \text{ g FLiNaK} \approx 65.43 \text{ g FLiNaK} \checkmark$$

Day 70 (Continued)

10/10/22

- Elektra / Canary Electrode Heights 1:1/3 Scale



- 1/2 in submersion (electrodes in FLiNaK)

Day 72

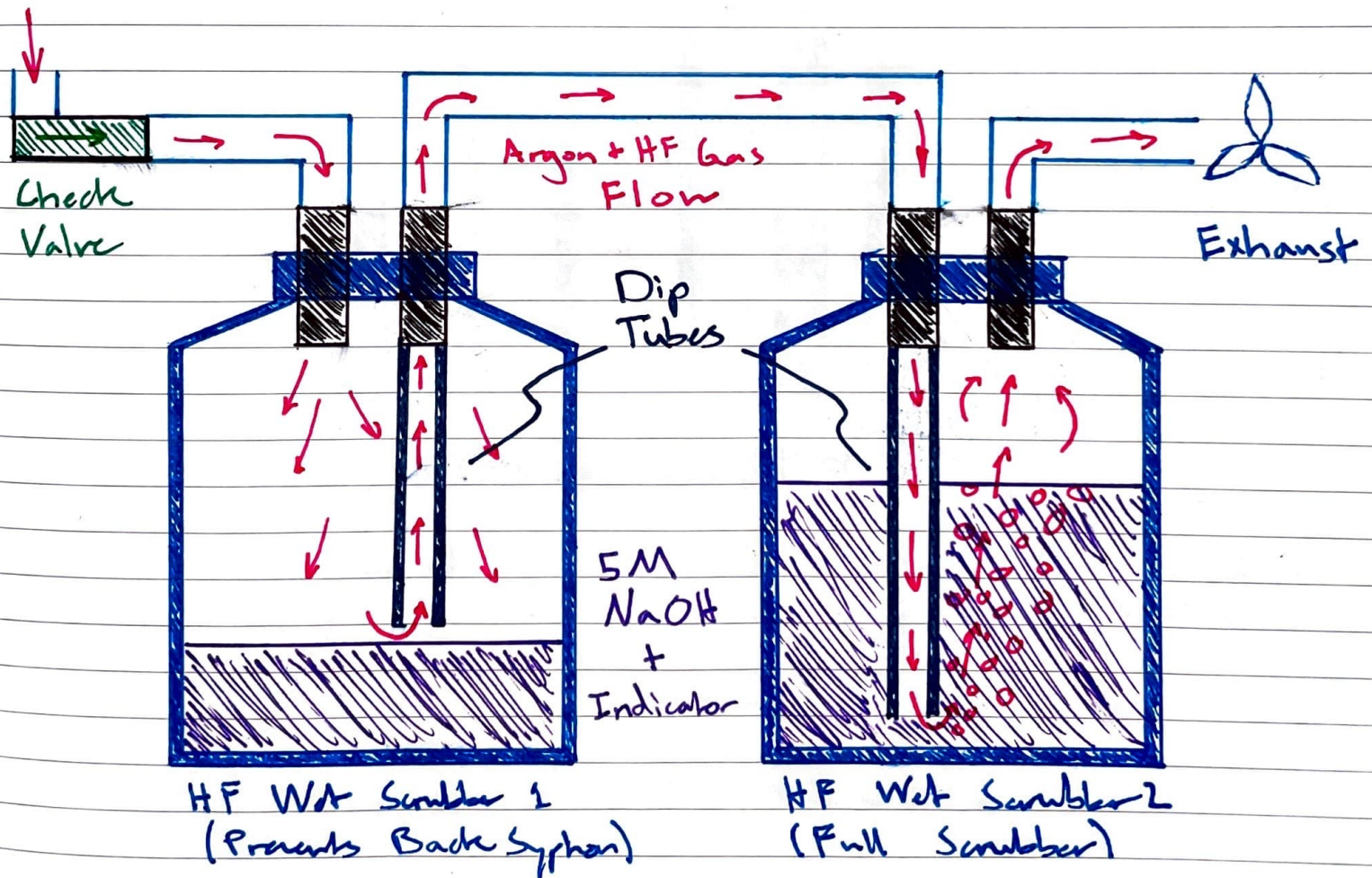
10/12/22

- Elektra turned on 10:00 AM, ramp 5°C/m to 65°C
  - Yesterday, I purposefully left the system unpressurized to test the back siphon phenomenon
- 5M NaOH + Indicator was pulled into the capture scrubber (only a small amount)

I am curious the pre-requisites for total scrubber pull... infinite vacuum?

I repressurized the system before major transfer, hopefully my salt is not contaminated...

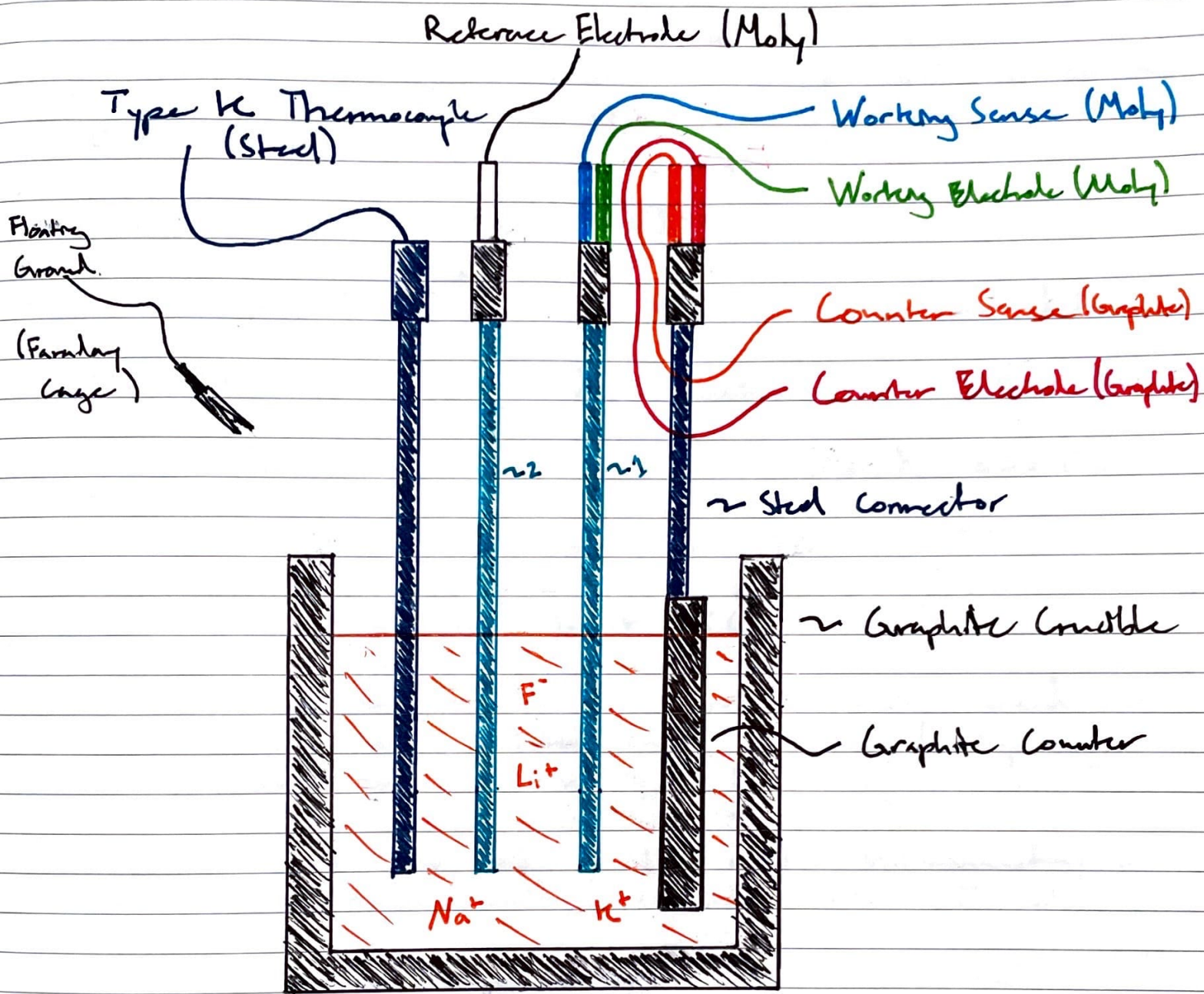
- Possible Syphon Control Scrubber System Redesign



Day 72 (Continued)

10/12/22

- Gamry Electrochemical Setup FLiNaK



- This electrochemical setup was utilized to isolate the redox potential window of Moly / FLiNaK

- 1.725 V  
Reducing  
Potassium

+ 0.350 V  
Oxidizing  
Molybdenum

Day 72 (Continued)

10/12/22

- OKR Completed <sup>(not really)</sup> ✓ Historic Moment!!! (kinda)

Develop reliable oxide measurements techniques for FLiBe by finishing Elektra setup and successfully running 10 <sup>CV</sup> SWV runs with gold electrodes in FLiNaK by 9/30/22.

/// Unfinished

/// Finished

- I collected first set of FLiNaK electrochemical data

2:00 P.M., PARR up + dip electrodes in salt

Hand held TC reader for salt showed 626.4°C

Furnace set point TC 650°C

Furnace limit TC 700°C

- Electrode heights seem to be perfect based off TC reading and electrochemical testing (perhaps single short or counter short... will verify in the future)

- Open circuit potential + 10 cyclic voltammetry runs with chronoamperometry cleaning after 5<sup>th</sup> CV

- Better electrode connections could be made, custom alligator clips for gold standard

- Many processes can be optimized to run Elektra

- Electrochemical parameters to be discovered like operating voltage window, scan rate prime, oxidation + reduction potentials

Day 78

10/20/22

- Formal procedure

- Adding lithium oxide, does not change level

- New batch for each lithium oxide

- Calculate based on mass of lithium oxide

- Lithium oxide melting point

- Avoid carry over error

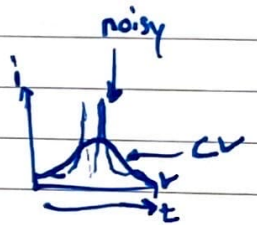
- Baseline already contaminated

- Solubility delay, dissolving problem

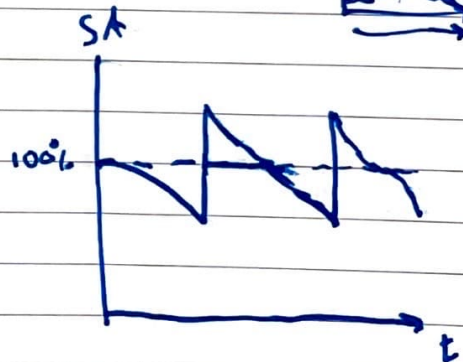
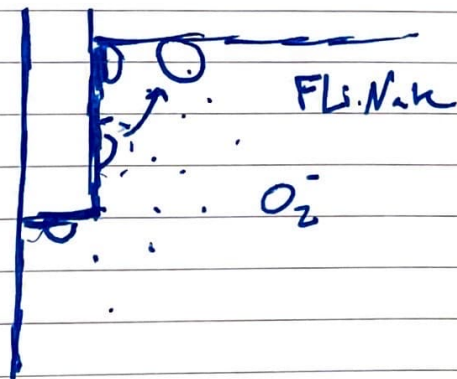
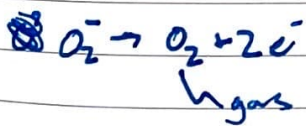
- Calculate oxygen atoms bubbled

- Explore square wave in any case

- Bubbles change surface area



① → GMdas Free Energy



Bubbles also adjust surface, enhances  $\text{O}_2^-$  diffusion, plays with correlation

Day 78

10/20/22

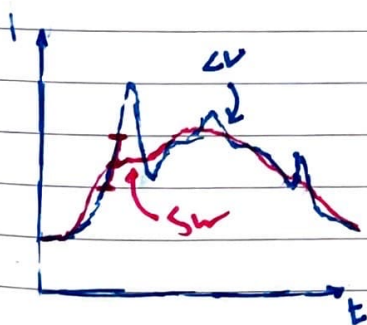
- Eventually measure surface area, gold electrode
- Impose concentration, calculate diffusion, measure oxide
- CV Randles Sevcik equation

$$[C][D][SA][TP][\text{constants}]$$

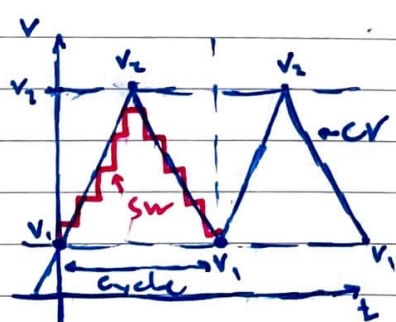
- current <sup>densities</sup> rate of dehydrogenation

(- current density makes independent from surface area  
 the current itself is dependent on surface area  
 (overall magnitude))

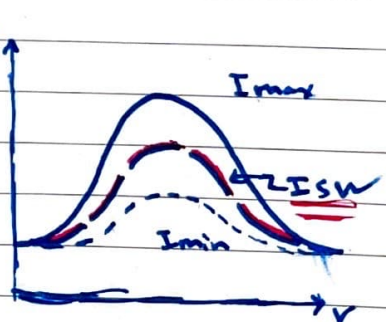
- Reduce electrode little current running through it
- Counter electrode ideally infinite
- Two weeks goal for ETU-ET (apply for miles)
- S-Lab ingot prep  $\downarrow$  ask Sam
- S-Lab graphite pellets



Cyclic Voltammetry  
 Averaged by  
 Square Wave



Potential Applications  
 Cyclic Voltammetry  
 Square Wave

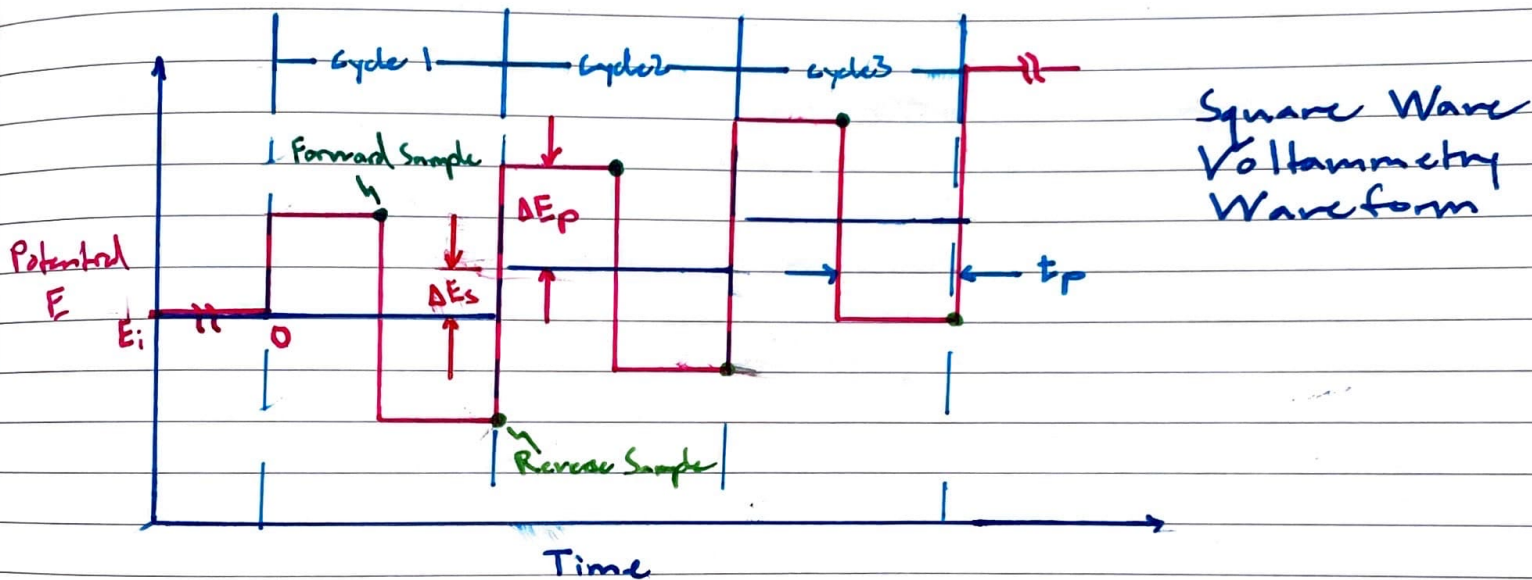


Square Wave  
 Current  
 Return



- Electrochemistry notes

- Square wave voltammetry invented by Ramsay and Krause further developed by Osteryoungs
- Combines the best part about pulse techniques
- Symmetrical double pulses, one forward one reverse



- No true polarization, forward + reverse currents applied

↳ not sure about this (nomenclature forward + reverse based on direction from staircase)

- $\Delta E_p$ , pulse height (mV)
- $t_p$ , pulse width, frequency (Hz)

... specifically  $f = 1/2 t_p$

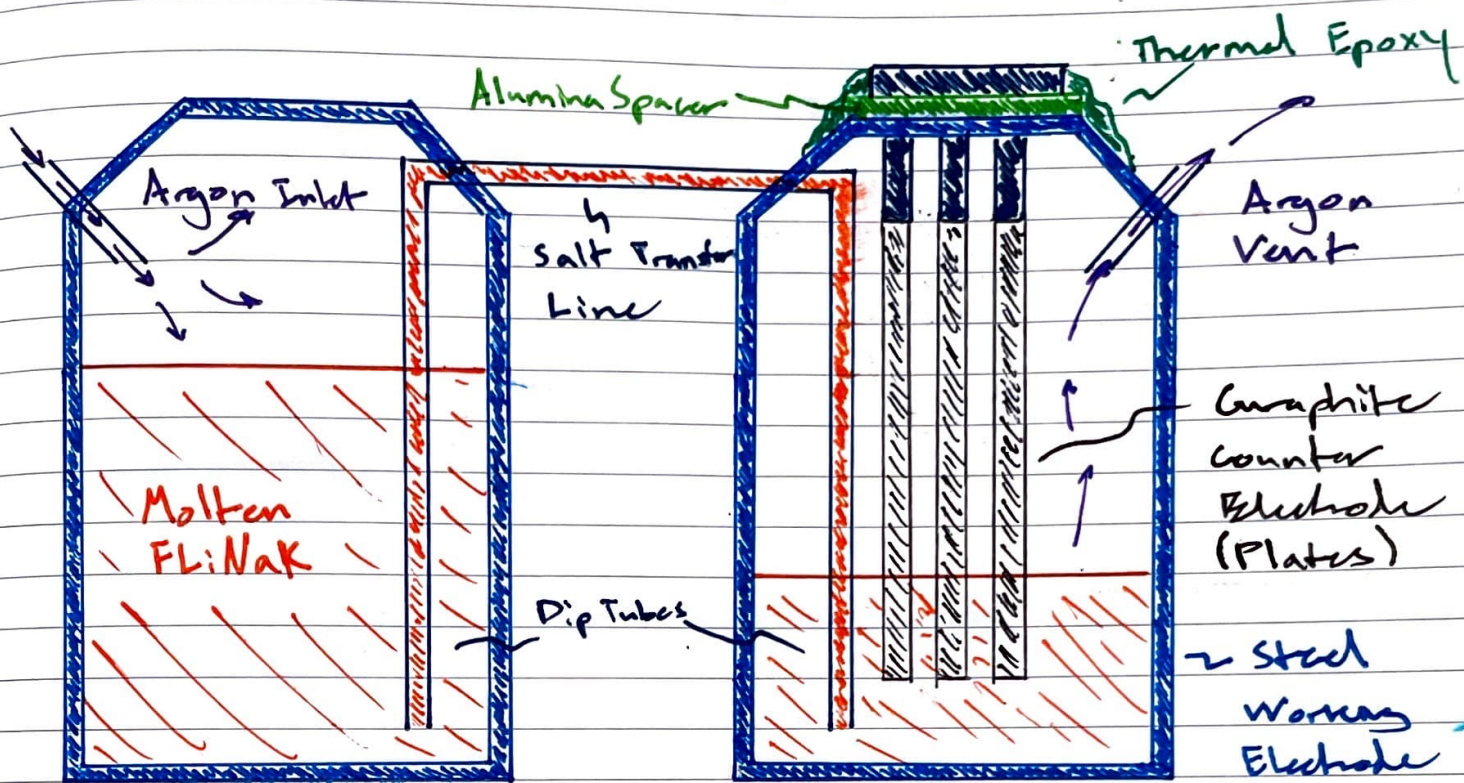
-  $\Delta E_s$ , stair-case shift beginning of each cycle

- Scan rate  $v = \Delta E_s / 2t_p = f \Delta E_s$

Day 89

11/7/22

## - FLiNaK Purification Design (PREV2)

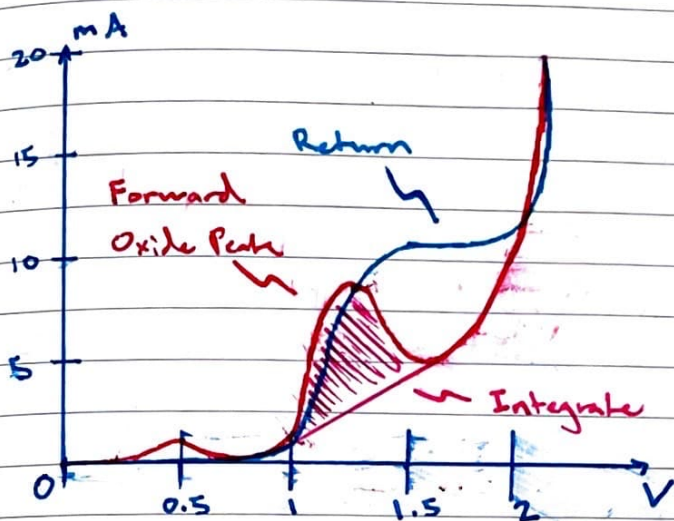


- An empty salt tank can be converted into a giant electrochemical cell
- Working electrode can plate out impurities
- Counter electrode will generate fluorine gas
- This would require massive amounts of power
- Once the salt is pure, it can be transferred back to the storage tank
- Raw sodium or potassium is highly explosive
- Alumina spacer ring isolates electrodes
- This is a difficult process to navigate

Day 89

1/7/22

The Amount of Charge Passed From Cyclic Voltammetry



- From an integration of the oxide peak based off an arbitrary baseline, we discover charge passed...

4.09 milli Coulombs

1 mC has the equivalent charge of...

$$6.2415 \times 10^{15} \text{ electrons}$$

$$\Rightarrow 4.09 \text{ mC} \cdot \frac{6.2415 \times 10^{15} \text{ e}^-}{1 \text{ mC}} = 2.52718 \times 10^{16} \text{ e}^- \text{ passed...}$$

To convert to mols utilize Avogadro's number...

$$2.52718 \times 10^{16} \text{ e}^- \cdot \frac{1 \text{ mol}}{6.022 \times 10^{23} \text{ units}} = 4.19658 \times 10^{-8} \text{ mol e}^- \text{ passed...}$$

To find mols of  $\text{O}^{2-}$  consumed and  $\text{O}_2(\text{g})$  generated...



$$4.19658 \times 10^{-8} \text{ mol e}^- \cdot \frac{1 \text{ mol O}_2(\text{g})}{4 \text{ mol e}^-} = 1.04915 \times 10^{-8} \text{ mol O}_2(\text{g})$$

$$4.19658 \times 10^{-8} \text{ mol e}^- \cdot \frac{1 \text{ mol O}^{2-}}{2 \text{ mol e}^-} = 2.09829 \times 10^{-8} \text{ mol O}^{2-} \text{ ions}$$

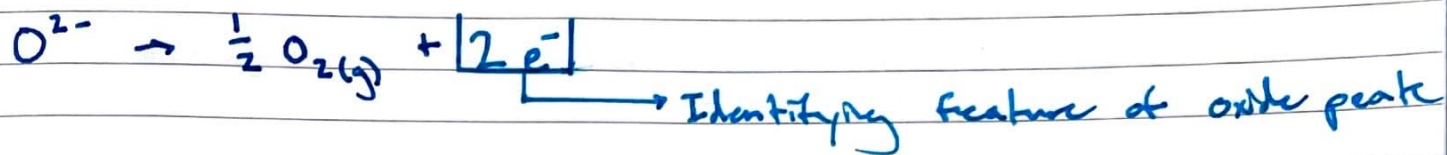
↳ Free Oxide Ions!!!

Day 10

11/8/22

- Identifying + Confirming Oxygen Peaks Gold Electrode

Mol ratios for oxygen from Massot...



From Massot as well...

$$W_{1/2} (\text{Descent Peak Half Width}) = RT/nF = \text{Volts or J/C}$$

$$\Rightarrow n = RT/W_{1/2}F$$

For Cyclic Voltammetry...

$$n_{cv} = \frac{(8.314 \text{ J/K mol})(923.15 \text{ K})^{(3.52)}}{(0.157 \text{ J/V})(96,485 \text{ C/mol})} = 1.761 \rightarrow \text{Close to } 2\text{e}^-$$

For Square Wave Voltammetry...

$$n_{swv} = \frac{(3.52)(8.314 \text{ J/K mol})(923.15 \text{ K})}{(0.217 \text{ J/V})(96,485 \text{ C/mol})} = 1.290 \rightarrow \text{Not so close}$$

- Heavy differences between calculated and predicted values come from arbitrary half peak widths...
- Specifically, the descent point is relatively unstable compared to the center, also functioning as maximum peak height.
- Further iterations and identical methods will most likely improve precision, accuracy, and a closer result ✓

Day 91

11/9/22

# - Calculating Free Oxide Concentration Cyclic Voltammetry

Randles Sevcik utilizes the curve peak...

$$i_p = 0.4463 n F A C \sqrt{\frac{n F v D}{RT}} \Rightarrow C = \frac{i_p}{0.4463 n F A \sqrt{\frac{n F v D}{RT}}}$$

Variables...

$i_p$ = Current Maximum (Amps)	0.004513
$n$ = Number electrons transferred redox event	2
$A$ = Electrode Surface Area ( $\text{cm}^2$ )	0.406834
$F$ = Faraday Constant (C/mol)	96,485
$D$ = Diffusion Coefficient ( $\text{cm}^2/\text{s}$ )	$4.74 \times 10^5$
$C$ = Concentration (mol/ $\text{cm}^3$ )	unknown
$v$ = Scan Rate (V/s)	0.25
$R$ = Inert Gas Constant (J/kmol)	8.314
$T$ = Temperature (K)	923.15

$$\Rightarrow C = \frac{0.004513 \text{ (A/s)}}{0.4463 (2) (96,485 \text{ C/mol}) (0.406834 \text{ cm}^2) \sqrt{\frac{(2)(96,485 \text{ C/mol})(0.25 \text{ V/s})(4.745 \times 10^5 \text{ cm}^2/\text{s})}{(8.314 \text{ J/mol})(923.15 \text{ K})}}$$

$$= 7.46212 \times 10^{-11} \text{ mol O}^{2-} \text{ ions / cm}^3$$

Convert to mols + grams in 65.43 g FLiNaK (31.917  $\text{cm}^3$ )...

$$7.46212 \times 10^{-11} \text{ mol O}^{2-} \text{ ions} \cdot \frac{31.917 \text{ cm}^3}{1 \text{ cm}^3} = 2.38 \times 10^{-9} \text{ mol O}^{2-} \text{ ions} \checkmark$$

$$2.38 \times 10^{-9} \text{ mol O}^{2-} \text{ ions} \cdot \frac{16 \text{ g Oxygen}}{1 \text{ mol Oxygen}} = 3.81 \times 10^{-8} \text{ grams O}^{2-} \text{ ions} \checkmark$$

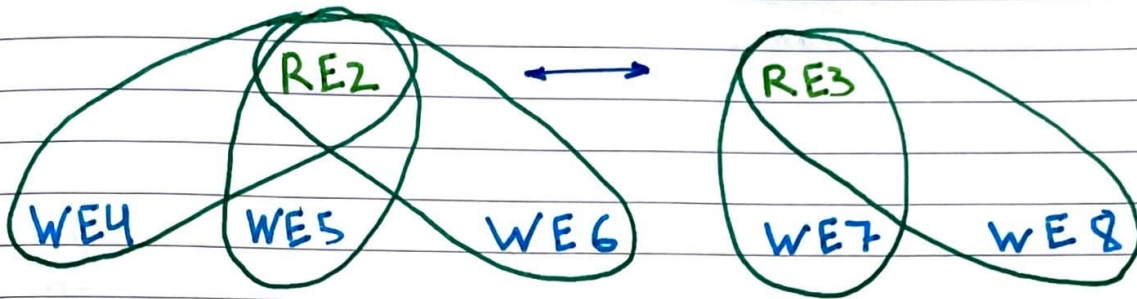
$$\text{ppm} = \frac{\text{Mass of Solute (g)}}{\text{Mass of Solution (g)}} \times 10^6 = \frac{3.81 \times 10^{-8} \text{ g O}^{2-} \text{ ions}}{65.43 \text{ g FLiNaK}} \times 10^6 = 0.000582 \text{ ppm} \checkmark$$

Day 92

11/10/22

- ETV 1.0 CCF Multiplexer Meeting with Rebecca

**CE1** - Direct from pstat to CCF



- Using only 1 potentiostat, for 2 CCFs  
... requires hardware switch for CE1

Option 1: Run wire from each counter to potentiostat, manually switch

Option 2: Figure out how to do it through MLTX

Option 3: Second multiplexer, switch between two channels

Can the counter sense follow the counter...

High positive, low negative, guard shielding  
↳ under use

Test connecting channels on different slot cards,  
enable us to copy Argon NL (National Labs)

Touch base with Sam, about ANL

Try to have a plan by end of next  
week, to finish wiring... check  
connections to multiplexer from pstat + electrodes

Day 93

11/14/22

### - Free Oxide Concentration Square Wave Voltammetry

Faulkner utilizes the current peak...

$$\Delta i_p = \frac{nFA\sqrt{D}}{\sqrt{\pi t_p}} \Delta \Psi \Rightarrow C = \frac{\Delta i_p \sqrt{\pi t_p}}{nFA\Delta \Psi \sqrt{D}}$$

Variables...

$\Delta i_p$ = Differentiated Current Peak (Amps)	0.004912
$n$ = Number electrons transferred redox event	2
$A$ = Electrode Surface Area ( $\text{cm}^2$ )	0.40684
$F$ = Faraday Constant (C/mol)	96,485
$D$ = Diffusion Coefficient ( $\text{cm}^2/\text{s}$ )	$4.74 \times 10^5$
$C$ = Concentration (mol/ $\text{cm}^3$ )	unknown
$t_p$ = Pulse Width (s) ~ Inverse Frequency	0.02778
$\Delta \Psi$ = Dimensionless Peak Limiter (Size: Resolution)	0.4531

$$\Rightarrow C = \frac{(0.004912 \text{ A/s}) \sqrt{(3.14159) (1/36 \text{ s})}}{(2)(96,485 \text{ C/mol})(0.40684 \text{ cm}^2)(0.4531) \sqrt{(4.74 \times 10^5 \text{ cm}^2/\text{s})}}$$

$$= 5.92501 \times 10^{-11} \text{ mol O}^{2-} \text{ ions / cm}^3$$

Convert to mols + grams in 65.43 g FLiNak ( $31.917 \text{ cm}^3$ )...

$$\frac{5.92501 \times 10^{-11} \text{ mol O}^{2-} \text{ ions}}{1 \text{ cm}^3} \cdot 31.917 \text{ cm}^3 = 1.89109 \times 10^{-9} \text{ mol O}^{2-} \text{ ions} \checkmark$$

$$1.89109 \times 10^{-9} \text{ mol O}^{2-} \text{ ions} \cdot \frac{16 \text{ g Oxygen}}{1 \text{ mol Oxygen}} = 3.02575 \times 10^{-8} \text{ grams O}^{2-} \text{ ions} \checkmark$$

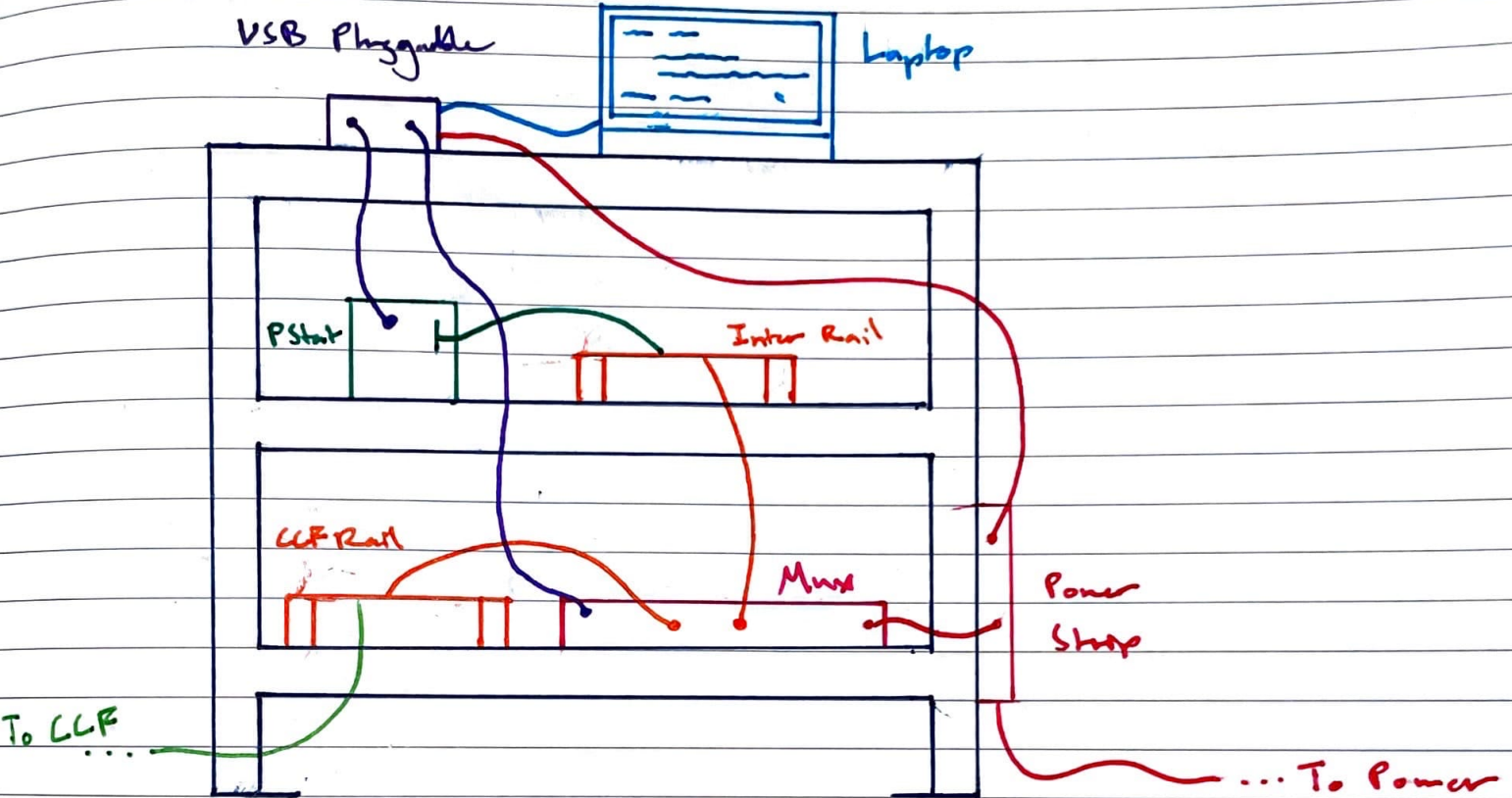
$$\text{ppm} = \frac{\text{Mass of Solute (g)}}{\text{Mass of Solution (g)}} \times 10^6 = \frac{3.02575 \times 10^{-8} \text{ g O}^{2-} \text{ ions}}{65.43 \text{ g FLiNak}} \times 10^6 = 0.00046244 \text{ ppm} \checkmark$$

CV  $\rightarrow$  0.000582 ppm      SWV  $\rightarrow$  0.00046244 ppm (Similar)  $\checkmark$

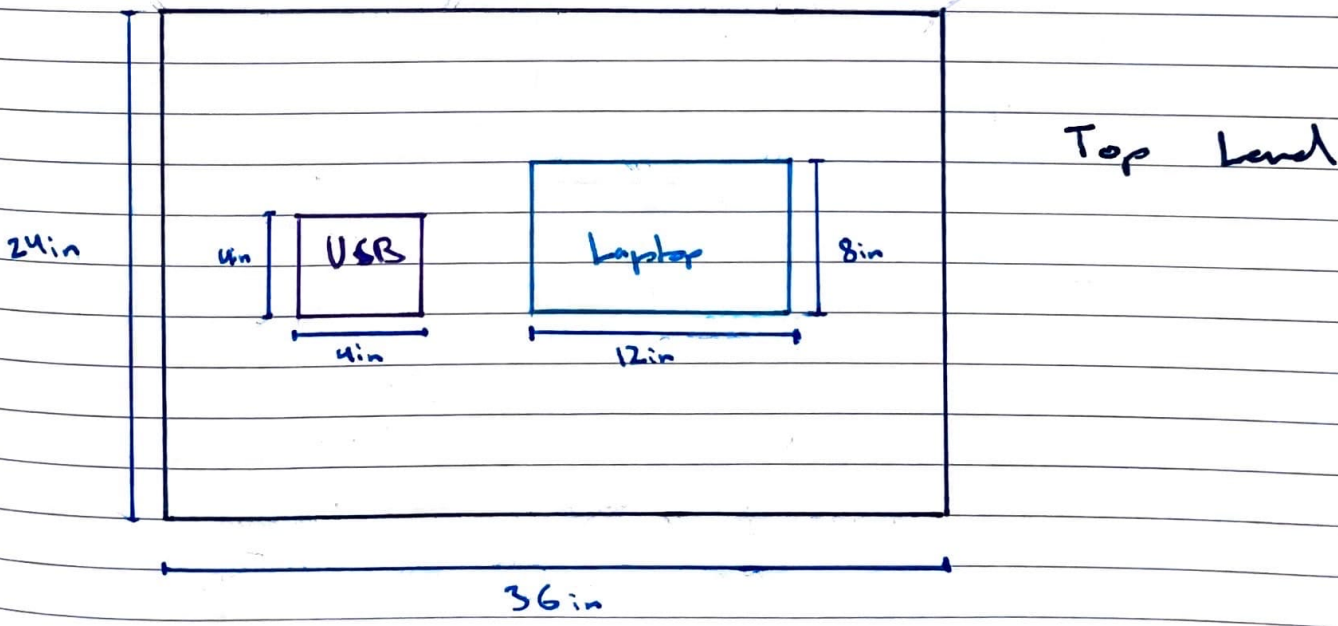
Day 100

11/23/22

- Multiplexer Arrangement for ETV LO LCF, 4:1 Scale



- Pathway Circuitry

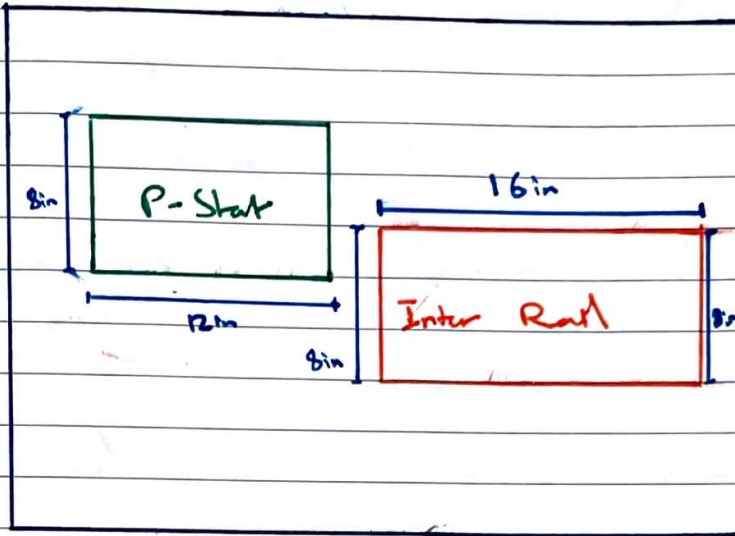




Day 101

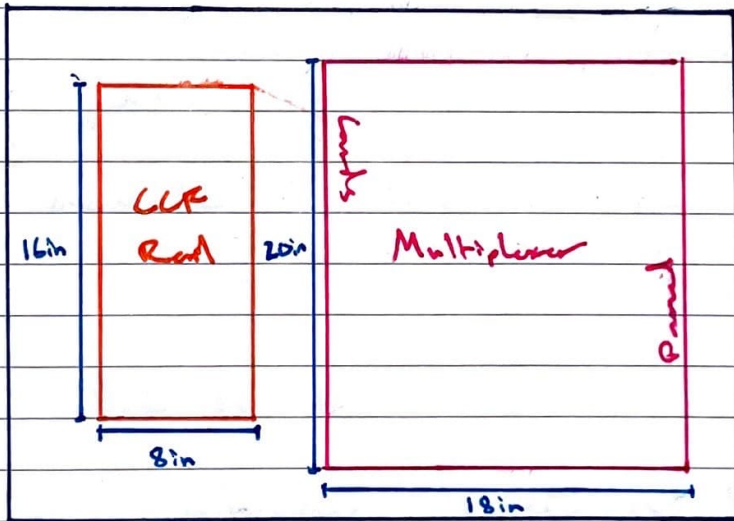
11/28/22

- ETV CCF Physical Setup Pstat / Mux Hit Scale



Middle Level

- Connections + Positions can be reconfigured



Lower Level

Multiplexer terminal cable connection resistances

Card 1: (30 ft) ETV	Channel	High	$\Omega$	Low	$\Omega$
	1	WE4	0.4 <del>RE2</del>	RE2	0.4
	2	WE5	0.4	RE2	0.3
	3	WE6	0.3	RE2	0.4
	4	WE7	0.5	RE3	0.4
	5	WE8	0.4	RE3	0.4
	6	RE3	0.5	RE2	0.4
	7	RE2	0.3	RE3	0.3
- Card 3: (30/40) ETV/IMS	1	CE1	0.4	~	~
	2	CE9	2.4	~	~
- Card 2: (40 ft) IMS	1	WE12	0.7	RE10	0.4
	2	WE13	11.7	RE10	0.4
	3	WE14	0.5	RE10	<del>X</del>
	4	WE15	0.5	RE11	0.4
	5	WE16	0.9	RE11	0.4
	6	RE11	0.4	RE10	0.3
	7	RE10	0.5	RE11	0.4

Measurements for the ETV LCF are pristine ✓  
 Low resistances encountered...  
 Stabilized around 0.4  $\Omega$   
 Linear sweep voltammetry calibrations are successful!

Some measurements for the IMS LCF are out of range... SP  
 The low for channel 3 (RE10) is a failure !!  
 The entire card was sabotaged by CE9 resistance drift. i-i

- Spent the weekend troubleshooting multiplexer
- Rechecked connections, formulas increase quality
- Card 3, channel 2, high CE9, measured high resistance  
 ↳ moved to channel 3, fixed problem ✓  
 (perhaps broken relay)
- Card 2, channel 1+2<sup>+5</sup>, highs WE12+13<sup>+16</sup>, measured high resistance  
 ↳ tightened connections, might have to move channels  
 (be careful when sliding in card)
- Card 2, channel 3, low RE10, no connection  
 ↳ found loose jumper on inter rail, fixed problem ✓
- Card 2, channel 1+2+5, high W12+13+16, high resistance  
 ↳ moved to channel 8+9+10 (switching card slots did not fix)  
 ... compared to previous

Channel	High	$\Omega$	Low	$\Omega$
1 → 8	WE12	0.3	RE10	0.3
2 → 9	WE13	0.4	RE10	0.2
5 → 10	WE16	0.3	RE11	0.3

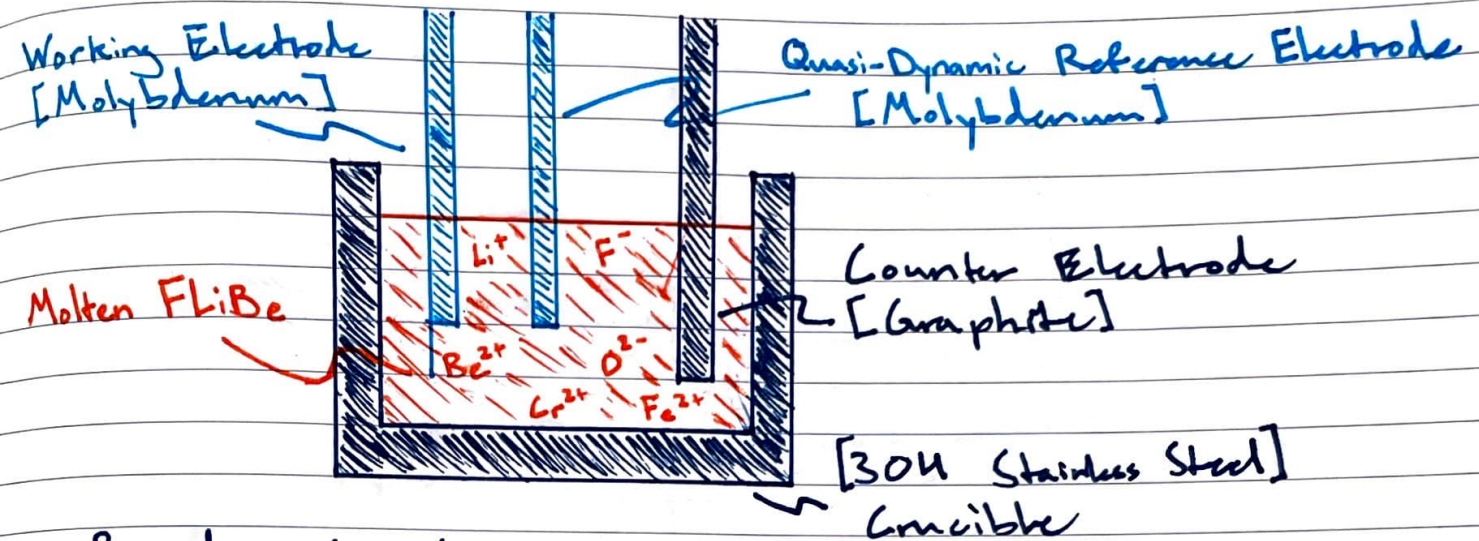
For card 2...  
 ... switching faulty  
 relay channels fixed  
 high resistances ✓

- I highly recommend purchasing additional cards for future repairs, modifications, or troubleshooting (broken pathway)
  - This will also increase the capabilities of the overall system ✓
- ↳ Unfortunately, this means some relays within MUX might be broken 😞

Day 110

12/12/22

## - ETU - ET Lanyary Representation



Render Simulation

## - Direct Current Electrochemical Test Methods

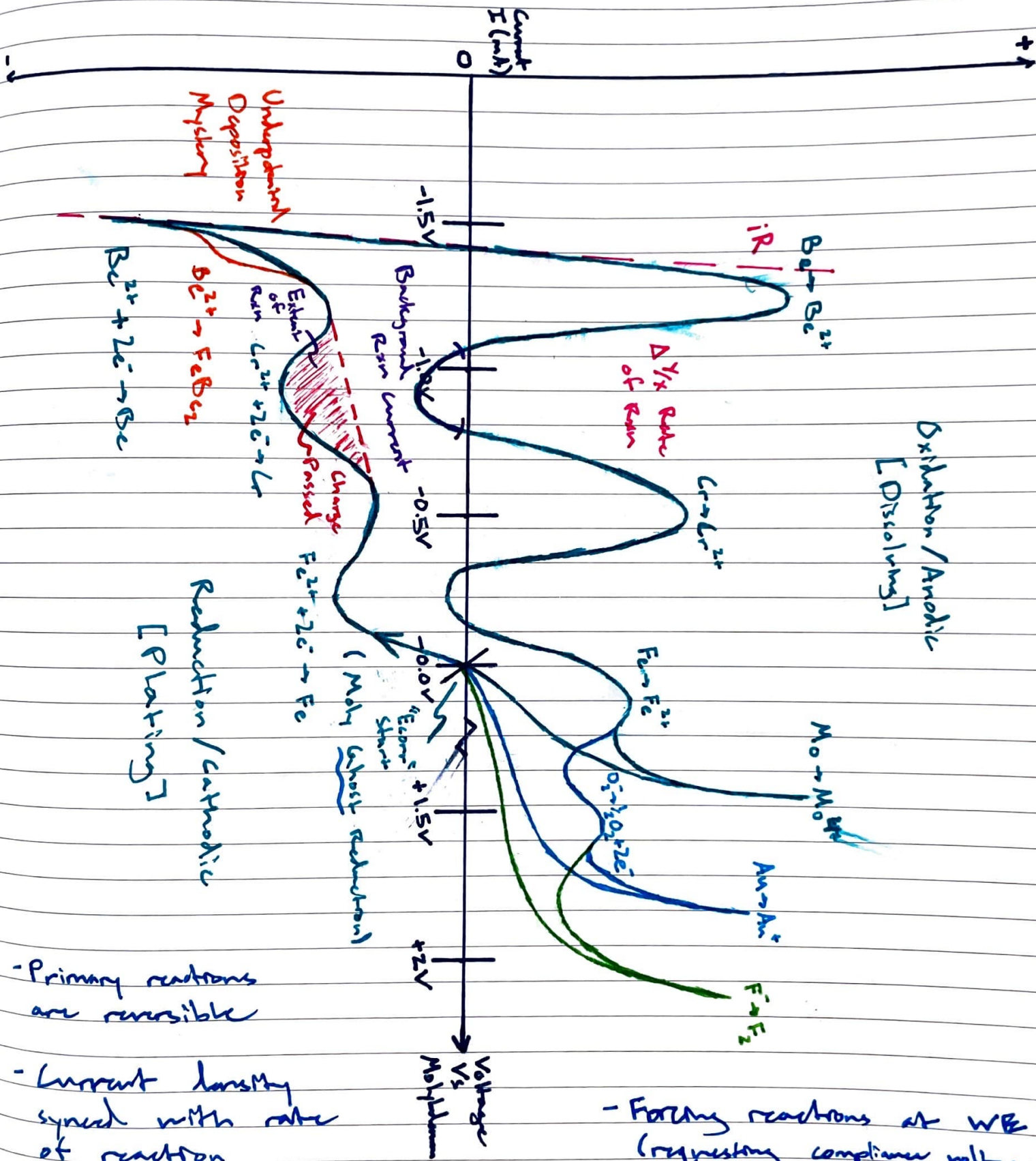
**Chronoamperometry:** Cleans working electrode by forcing exterior layers to dissolve

**Chronopotentiometry:** Plates working electrode with beryllium, then redissolves naturally, measuring redox potential

**Cyclic Voltammetry:** Plates multiple layers on the working electrode, then redissolves, establishing stability windows, extent of reactions, and rate of reactions

- Essentially forcing chemical reactions at the working electrode

FLiBe Electro Chemistry Visualized Cyclic Voltammetry



- Primary reactions are reversible

- Current density synced with rate of reaction

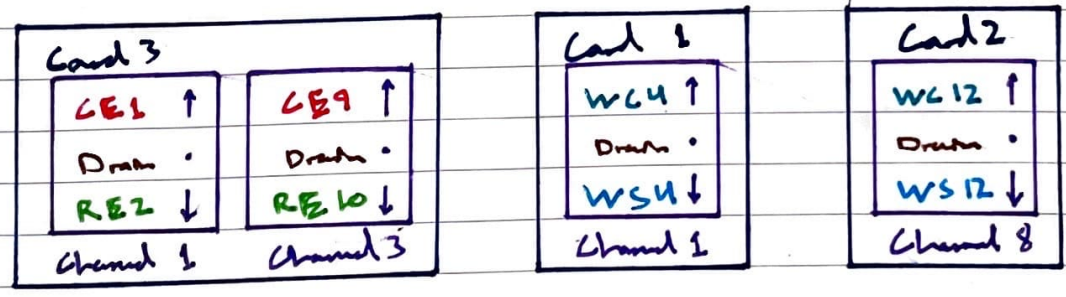
- Forcing reactions at WE (requiring compliance voltages)

- The objective for today is to test splitting the working wires and sense signals
- The temporary wired setup:

C1c1 C3c1

C2c2 C3c3

- Action required to connect system
  - Add REZ to low of C3c1 ✓
  - Add RELO to low of C3c3 ✓
  - Split WE4 on C1c1 to ↑ WC4 and ↓ WS4
  - Split WE12 on C2c2 to ↑ WC12 and ↓ WS12
  - Adapt wiring + connections as needed
- Simplified card schematic - kitted diagram



- Electrochemical testing through multiplexer conducted
  - ↳ similar to format as before
- Sequence: OCP → Oxidation → Reduction → Chronoamp → CV (window)

Day 12

12/14/22

- ETU 1.0 LCP Multiplexer Redesign ~~Shopping List~~ <sup>Wish</sup>



Wires + Cables: Long shielded cable 40 ft  
 $(\times 15 \times 2 + 5) \times 40 \text{ ft} = 1,400 \text{ ft}$

Intermediate shielded wire 4 ft  
 $(\times 20 \times 2 + 5) \times 4 \text{ ft} = 180 \text{ ft}$

Rail Terminals: Black for shield  
 $(\times 30 \times 2 + 5) = 65 \text{ terminals or units}$

Blue for WE designators  
 $(\times 5 \times 2 + 5) = 15 \text{ units}$

Green for RE designators  
 $(\times 2 \times 2 + 5) = 9 \text{ units}$

Red for CE designators  
 $(\times 1 \times 2 + 5) = 7 \text{ units}$

Grey for signal splitters  
 $(\times 10 \times 2 + 5) = 25 \text{ units}$

Connections: Terminal Jumps 10 long  
 $(\times 5 \times 2 + 5) = 15 \text{ units}$

Formulas for Pad Connections  
 $(\times 200 \times 2 + 100) = 500 \text{ units}$

Keithley High Count Leads  
 $(\times 3 \times 1 + 3) = 6 \text{ units}$

Electrode Connector Rings  
 $(\times 8 \times 2 + 10) = 26 \text{ units}$

Day 113

12/15/22

- Today some interesting ideas were covered and discovered regarding ETU LO GF multiplexer design
- Specifically, it seems optimal to setup the Max IMC so...

Card 1: ETU + IMS Counter / Reference

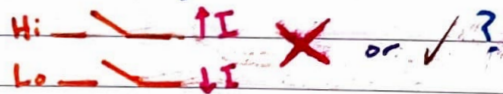
Card 2: ETU Working Current / Sense

Card 3: IMS Working Current / Sense

- The above design is...

- The most efficient setup ✓
- Allows card space for backups ✓
- Enables the maximum maneuverability / adaptability ✓
- Requires the least amount of physical wiring (reduces opportunities for failure) ✓

- High current applications still need to be tested through double relays



→ Use SS GE to produce  $F_2/F_4$  (or perhaps even aqueous chemistry)

- Alternative designs consider housing the current and sense signals on different cards altogether, however these methods constrain us on many other different levels
- Similar problems arise when comparing the approach of Argonne National Laboratory, distinct problem statement altogether

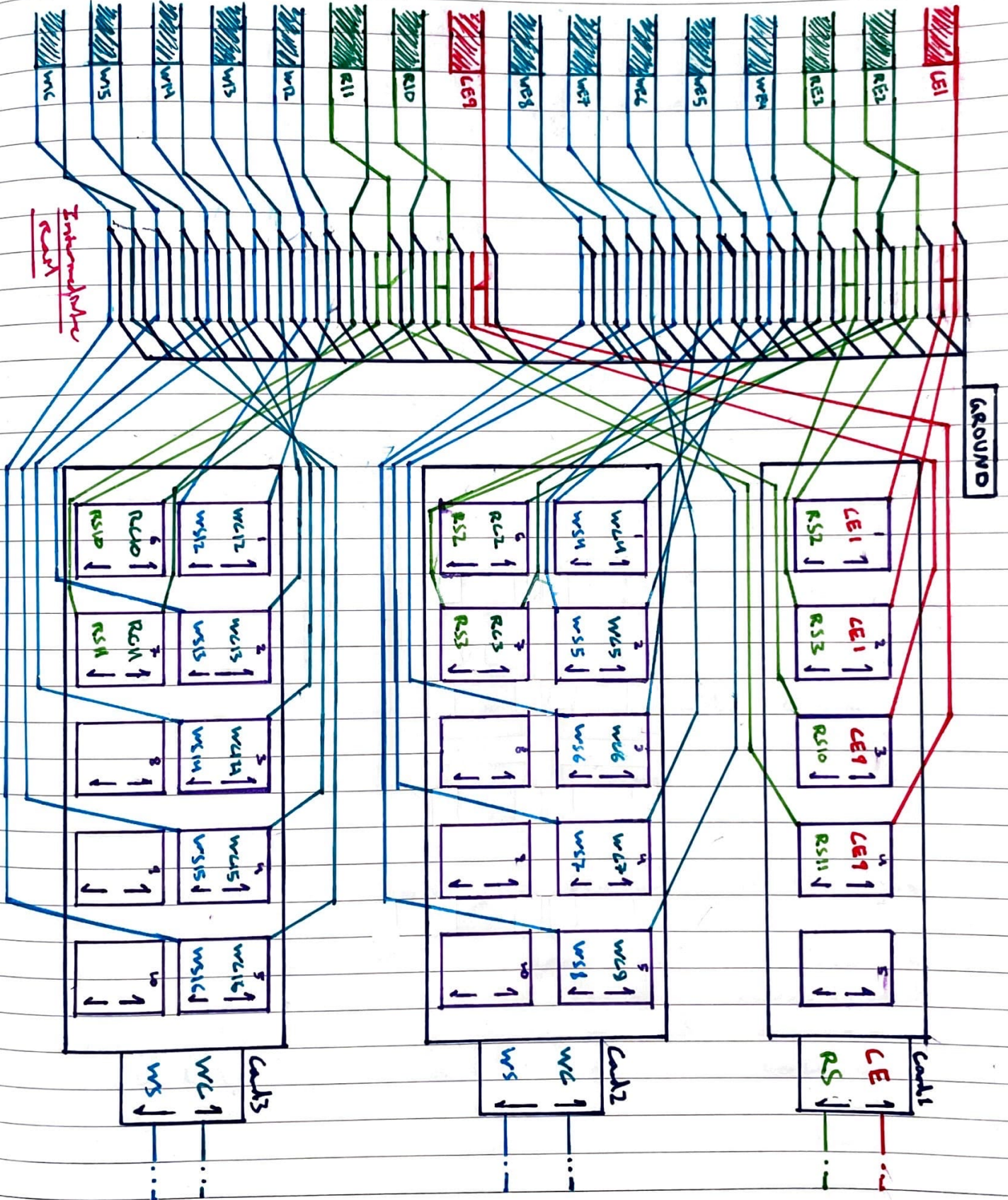
High current testing could include negative 2 A chronopotentiometry and studying various electrode reactions... Impurities +  $C^+$   $CF_x + F_2$  ??? (Argonne)



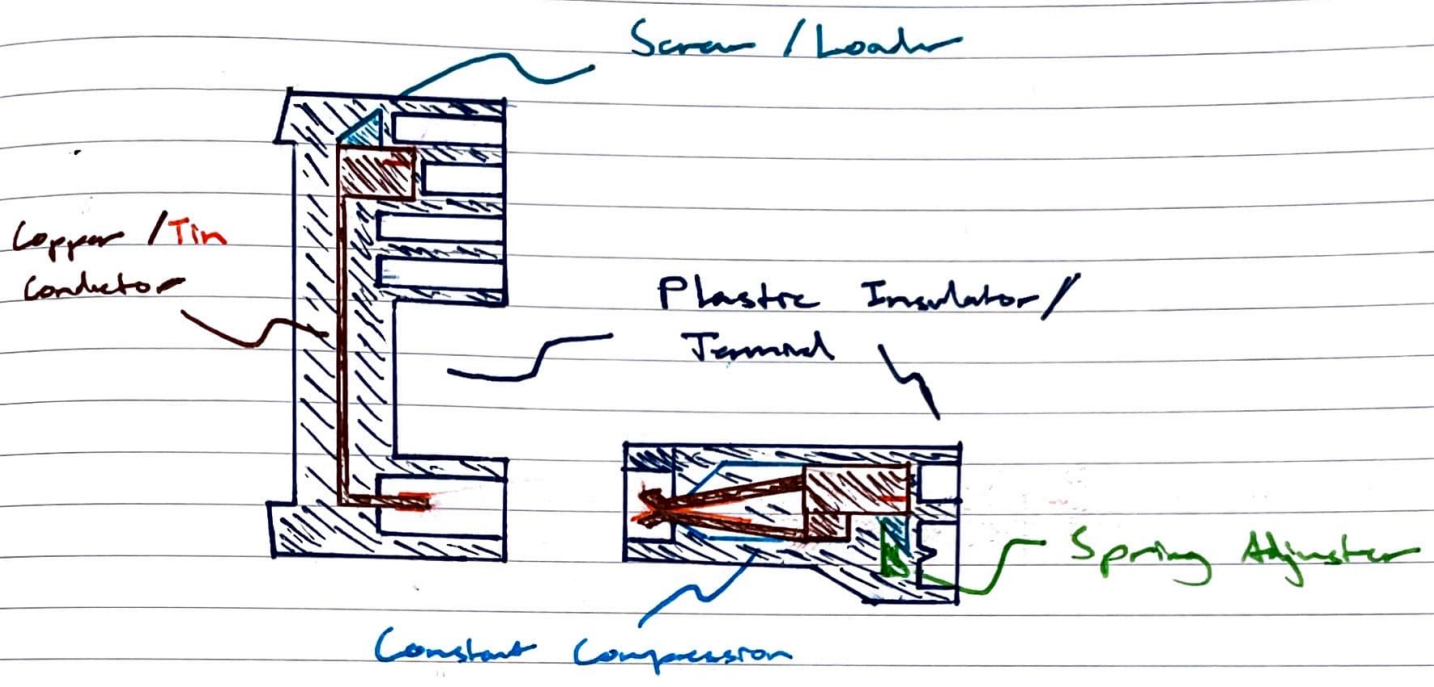
Day 114

12/16/22

# - ETU 1.0 CLP Multiplexer Redesign Mk III



- Terminal Block Plug & Play Sketch



- A new kind of terminal connection was discovered
- Pluggable terminals can be attached to a din rail
- Incorporating these terminals will...
  - Enable ultimate levels of versatility/adaptability
  - Eliminate the need for operator screws (simple system to change)
  - Efficiently assemble & disassemble setup
- Possible failure points increased downside
- Resistance compensated through potentiostat (sense pathway low current, negligible resistance)

Day 117

12/21/22

- Multiplex Relay Precision Resistances

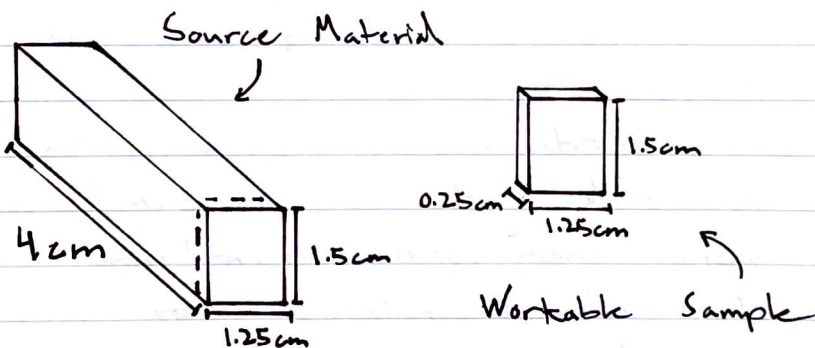
Channel	Highs ( $\Omega$ )			Lows ( $\Omega$ )		
	Card 1	Card 2	Card 3	Card 1	Card 2	Card 3
1	0.07	0.14	0.10	0.09	0.14	0.16
2	0.08	0.42	0.43	0.08	0.06	0.15
3	0.07	0.19	0.13	0.15	0.27	0.27
4	0.23	0.15	0.10	0.09	0.16	0.30
5	0.17	0.25	0.15	0.09	0.15	0.09
6	0.18	0.43	0.21	0.03	0.20	0.26
7	0.12	0.04	0.22	0.10	0.06	0.18
8	0.11	0.11	0.15	0.09	0.08	0.17
9	0.21	0.21	0.19	0.16	0.09	0.28
10	0.14	0.08	0.11	0.00	0.11	0.11

- Fluke 1507 insulation tester ~~was~~ utilized above
- Multimeter had to be rezeroed several times due to potential drift
- Although all ohmage seems fine, previous tests correlate with resistance peaks here, possible scaling
- Applies instrumented seemingly 5.25 V

Cutting

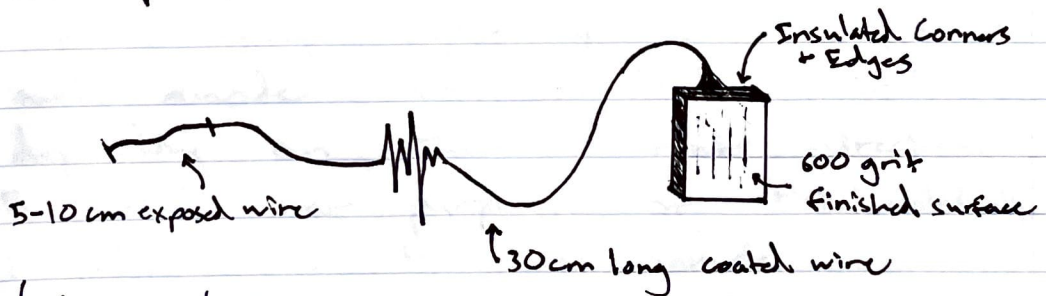
8/10/21

The first step in this electrochemical testing process is cutting raw material into workable sample sizes. Although other materials were already nicely formed into electrode shapes, Nitromax had to be cut. The Nitromax sources were all similarly shaped, 4 cm centimeter by 1.5 cm by 1.25 cm, resulting in a testable area of  $1.875 \text{ cm}^2$ .



Nitromax is an extremely hard metal alloy. This made the cutting process quite difficult, especially considering that only slow and precise methods could be used in order to avoid altering the heat treatment and destroying the sample.

Green liquid electrical tape developed by Gardner Bender was the optimal insulating material for this experiment. It is bright green, which makes it easy to identify and notice on small work areas. It is thick, preventing dripping and drooping, and it dries quickly. However, ~~it can be~~ the fumes that come off it can be nauseating. I recommend working outside or in a well ventilated area. I covered corners and edges to eliminate electromagnetic field concentrators. A wire coating length of about 30 cm is sufficient, as long as there is some exposed wires at the end to connect to the potentiostat.



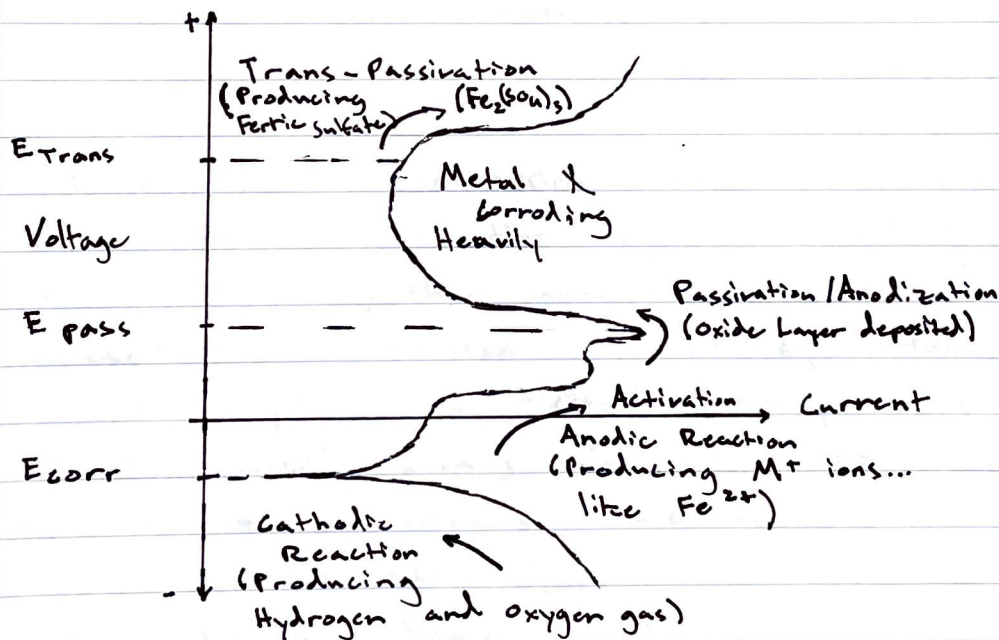
(Complete Electrode)

- M. S. Elasaad

Testing Continued

8/25/21

I retested the  $\times$  samples I did yesterday. Visually the data seems to be congruent for Nitromax Base Unknown, Norem O2, and Norem S8. Below is a sketch for a typical steel current-voltage curve.



As can be seen above, there are three distinct potentials that mark ~~more~~ relevant reactions in this linear voltage sweep.

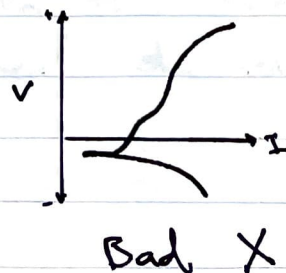
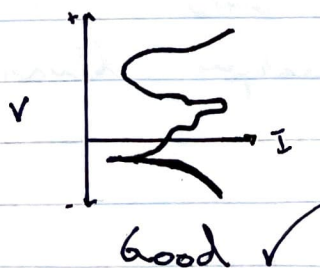
Even More Re-Testing

9/13/21

Today I got a large chunk of re-testing done as well as adding another Norum 02 sample to the data set.

I am encountering many problems and am discovering many solutions. First, an uneven polish ruins data. By this I mean, a sample with multiple polishes on its surface (different grits). The difference in depth results in pitting and ruins the sample and data. If a sample is polished, the entire surface needs to be completely polished.

As mentioned before, physical interference can throw off data and even the test itself. Rather than experiencing all parts of the corrosion curve, the sample goes straight from the cathodic reaction to pitting.



Solutions & Reflections9/17/21

I have completed all the necessary testing needed for analysis and reportation. I have completed the majority of the planned testing. In terms of Nitromax, I have collected robust repeatable data for H48 and H52 both at  $1050^{\circ}\text{C}$  and  $1200^{\circ}\text{C}$ . Professor Smith will help me with analysis and assembling a report as well as a poster for presentation.

In terms of final notes on test methods, replacing the electrolyte with fresh solution seemed to help with reference overload. The professor suggested the electrolyte may have been contaminated with too many free ions. I deduce this is correct. Voltage start issues still occur (only when the cell is enabled). Tinkering with test settings did not help. Overall, I dedicated much of my life this summer into achieving and learning what I have. I look forward to future projects.

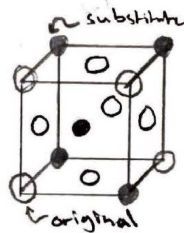
- M. G. Ghann



2. a) Solid solution strengthening utilizes a single phase while precipitation strengthening is multiphase.

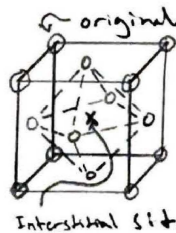
There are two types of solid solution strengthening. These are substitutional and interstitial. Both types involve editing the unit cell of a crystal lattice.

Substitutional replaces individual atoms in a unit cell with an alloying element of similar size. This creates small strains in the unit cell because the alloying element is slightly smaller or larger than the original element.



Here is the FCC unit cell with substitutional solid solution strengthening. The light atoms are the original element and the dark atoms are the slightly larger or smaller alloying elements.

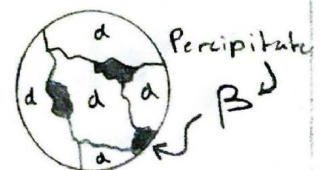
Interstitial inserts individual atoms of the alloying element into interstitial sites between the original atoms of a unit cell. This cramming act creates high strain within the lattice, much higher than substitutional, thus increasing strength.



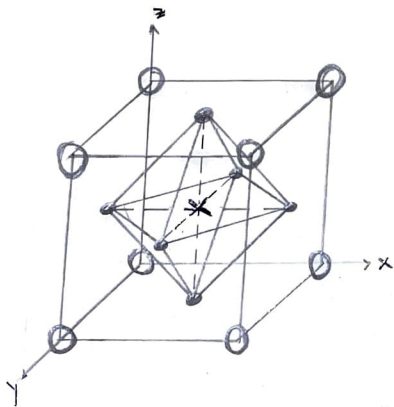
Here is the FCC unit cell displaying an octahedral interstitial site as an X. The original atoms are light circles. The outline of the atoms that touch the interstitial atom forms an octahedron in the FCC unit cell.

Precipitation strengthening occurs on a much larger scale. Now we are talking about different phases in the microstructure. This is a heat treatment. First a sample is heated up to a single phase temperature. Then it is quenched to keep the single phase at room temperature. Last it is naturally aged at room temperature or artificially aged at an elevated temperature. During aging, precipitates nucleate at grain boundaries and vacancies. These precipitates grow over time at certain temperatures which hinders dislocation movement, strengthening the sample.

Here is the microstructure for some generic precipitation strengthened alloy.



# Lead Zirconate Titanate



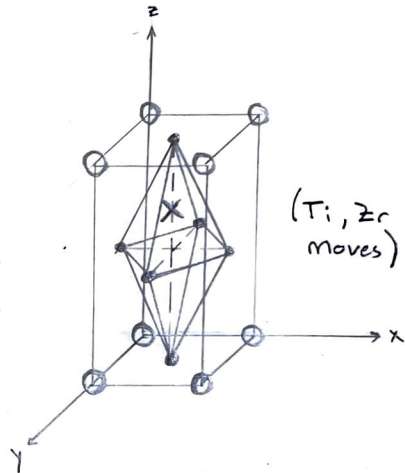
$\bigcirc = \text{Pb (Lead)}$

$\bullet = \text{O (Oxygen)}$

Increase  
Pressure



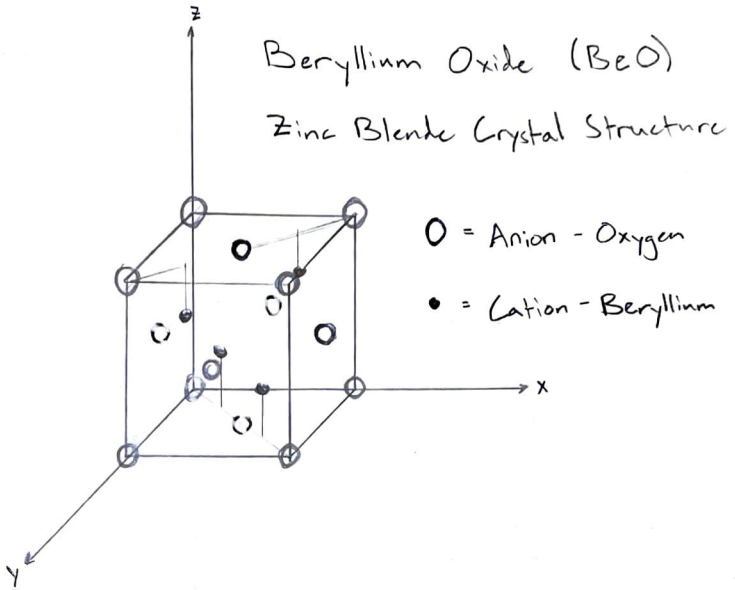
Increase  
Voltage



(Ti, Zr  
moves)

$\times = \text{Ti, Zr (Titanium, Zirconium)}$

1.



2.

[Lattice] + [Basis]

Cubic F + 2 atoms / Lattice Point

Beryllium cations are  $\frac{a_0\sqrt{3}}{4} \langle 111 \rangle$

from oxygen anions.

3.

Frenkel Defect

