Exp. No. 12	Experiment/Subject Ion Exchange Separation	Date 3/19/20	
Name	Lab Partner	Locker/ Desk No.	Course & Section No. 118

Objective: Determine the concentration of Nickel and Cobalt in an unknown solution using ion-exchange separation: Nickel will be complexed to form a red-brown complex, and Cobalt will be converted to blue $Co(CNS)_4^{2-}$; both will be measured spectrophotometrically

Theory

Ion exchange equilibria: treat with law of mass exchange.

General: for resin and aqueous ion A^{x-}

$$x\{RN^+(CH_3)_3\,Cl^-\}(resin) + A^{x-}(aq) \rightleftharpoons \{[RN^+(CH_3)_3] \times A^{x-}\}(resin) + xCl^-(aq)$$

- or -

$$x[(res^+)\,Cl^-] + A^{x-} \rightleftharpoons x\,Cl^-(aq) + [(res^+)\,A^{x-}]$$

for which $$K = \frac{[(res^+)^x A^{x-}][Cl^-(aq)]^x}{[(res^+)\,Cl^-]^x\,[A^{x-}(aq)]}$$

For $CoCl_4^{2-}$ ion and basic ion exchange resin $\phi - CH_2N^+(CH_3)_3\,Cl^-$

$$CoCl_4^{2-}(aq) + 2[\phi - CH_2N^+(CH_3)_3\,Cl^-](resin) \rightleftharpoons 2Cl^-(aq) + 2[\phi - CH_2N^+(CH_3)_3 - CoCl_4^{2-}](resin)$$

with large K such that $CoCl_4^{2-}$ is retained on the resin @ low pH.

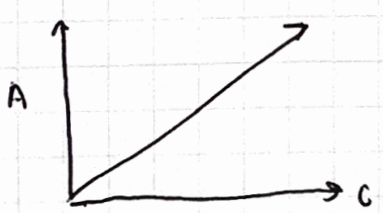
ions like Ni^{2+} that do not complex with Cl^-, K is small and Ni^{2+} is not retained (thus separating the Ni from the Co)

* Ni red complex: oxidize Ni w/ bromine in ammoniacal solution + treat with dimethylglyoxime (measure @ 450 nm)
* Co blue complex: convert metal to $Co(CNS)_4^{2-}$ in acetone/ethanol solution to prevent complex dissociation in H_2O (measure @ 625 nm)

Beer-Lambert law

$A = \varepsilon C l$

$y = m x$



The introduction/goals/theory section should be ~1 page for an in-person experiment and ~3 pages for an online lab

Signature sign	Date date	Witness/TA	Date

Exp. No. 12	Experiment/Subject Ion-exchange separation	Date 3/19/20	
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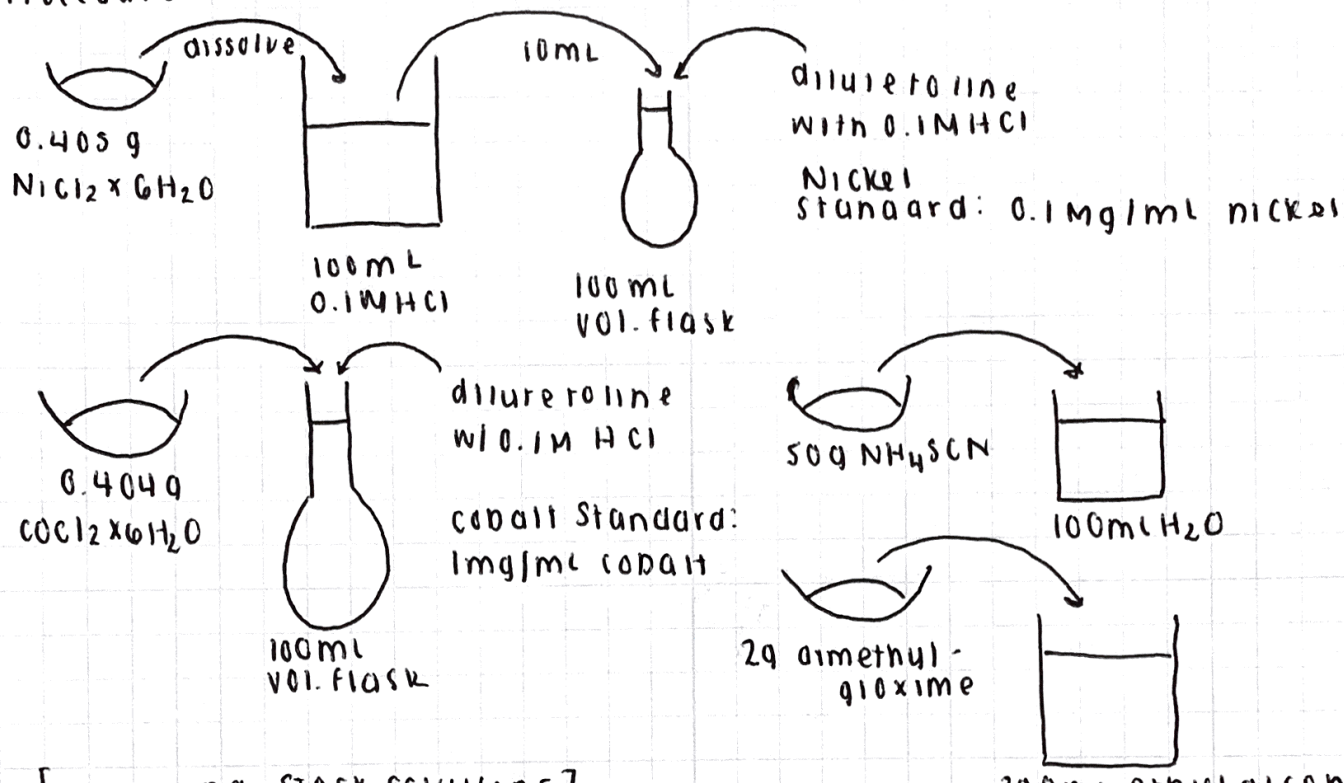
Your flow chart or abbreviated steps only need to be as detailed as you need to perform the experiment by using them without having to consult the experimental writeup.

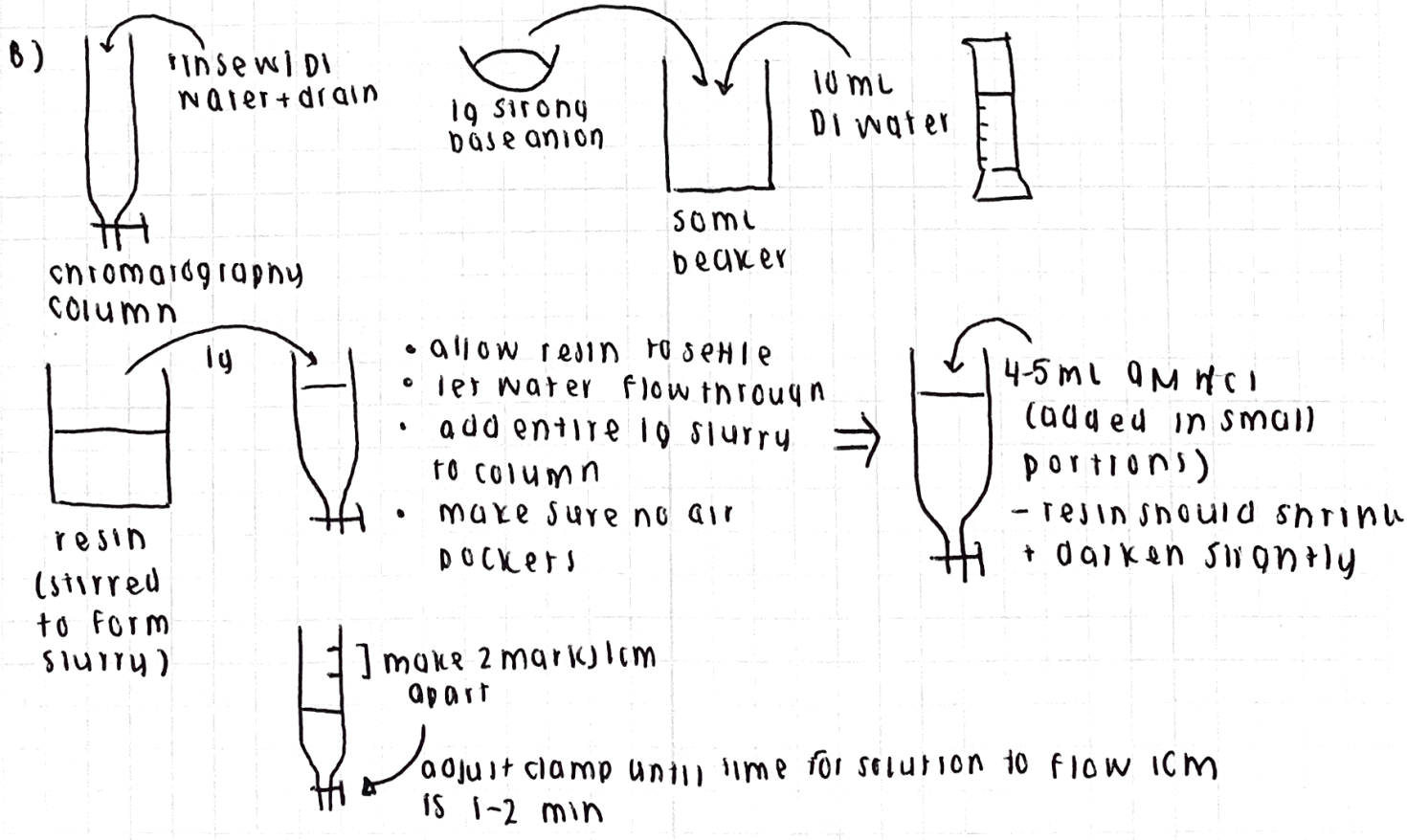
Procedure

A)



[preparing stock solutions]

B)



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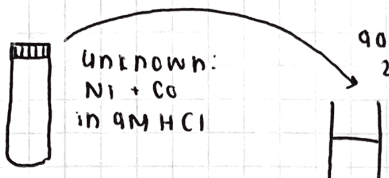
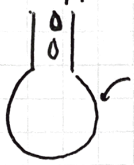
c)

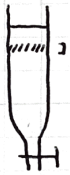


add 0.2 mL with 250 uL pipettor

- put tip as close to resin bed as possible
- add solution as slowly as possible



clean 250 mL volumetric flask



solution seeps into resin
- blue (cobalt) band
- will spread during washing but will not come off column

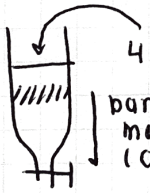
4 1-mL portions 9M HCl (allow each to settle before adding next portion)

end of last wash

do not scratch out

catch ~~1~~ drop in beaker

add
1 drop concentrated NH_3
+ 1 drop dimethyl glyoxime
- no red color should form



4-5 1mL portions 1MHCl

band should move down + into flask
(color blue first, then pink)

Ni flask
(set aside)

50mL vol. flask

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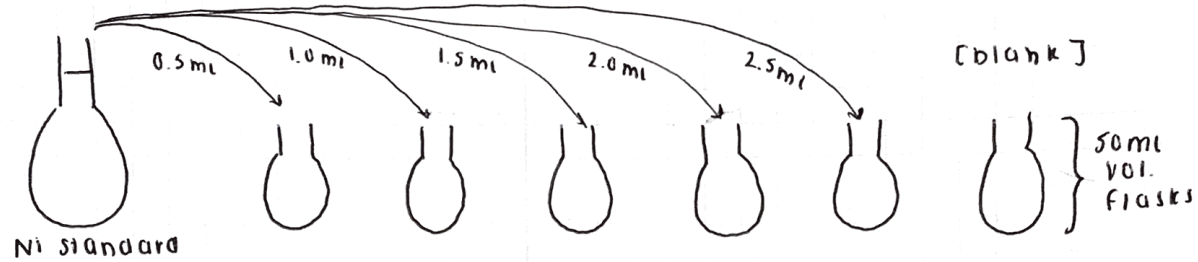
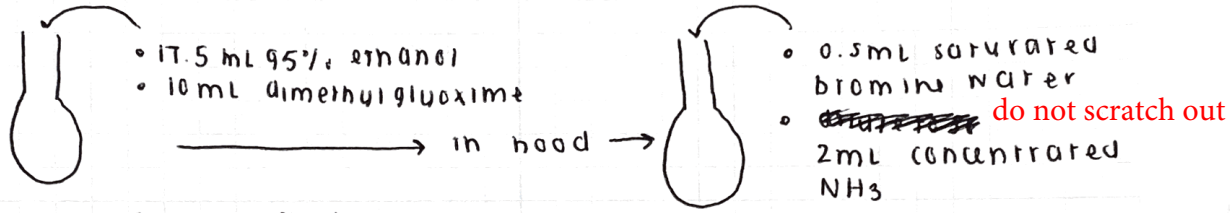
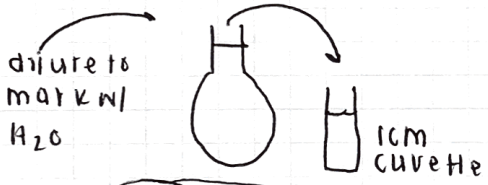
D)



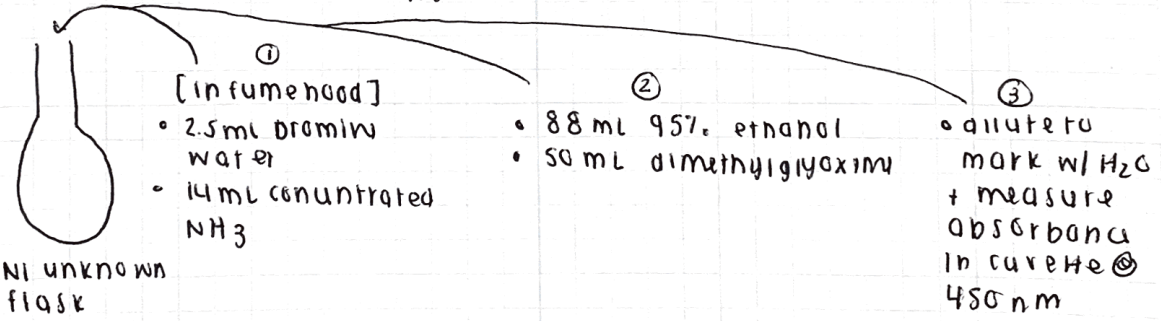


do not scratch out

* to each standard flask



- measure absorbance of each solution, using blank as reference (can wait more than 30 min)
- measure @ 450 nm



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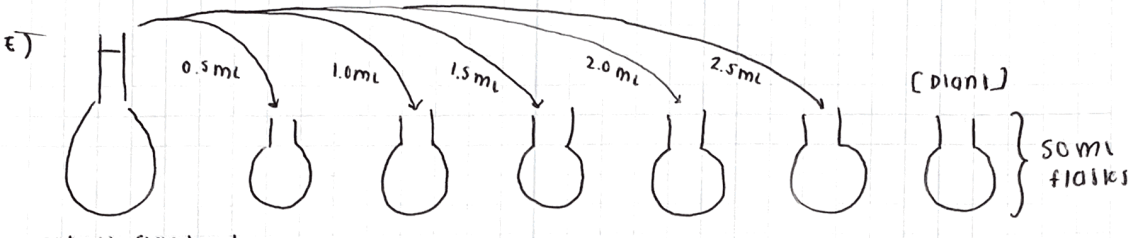
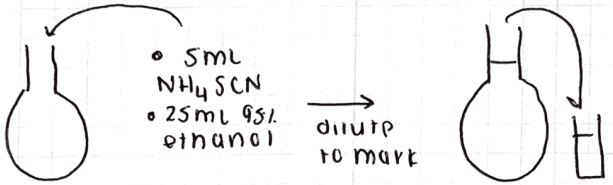
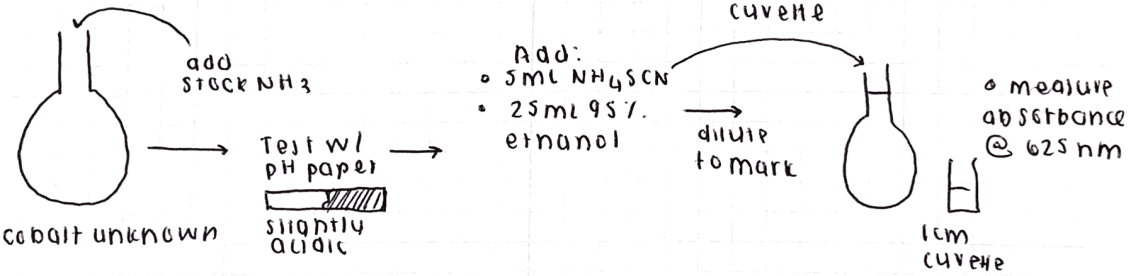
Nam	Lab Partner	Locker/ Desk No.	Course & Section No. 118

E)



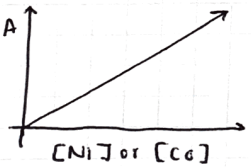
TO EACH FLASK:





F) Calculations

plot graph of A vs concentration for Ni, Co standard solutions



using least squares method

$A = \underset{y}{\varepsilon} \underset{m}{l} \underset{x}{c}$

→ use to calculate [Ni] and [Co] from unknown

calculate uncertainty:

$$u_x = \frac{s_y}{|m|}\sqrt{\frac{1}{k}+\frac{1}{n}+\frac{(y-\bar{y})^2}{m^2\sum(x_i-\bar{x})^2}}$$

where n, s_y, m, x_i, $\bar{x}$, $\bar{y}$ refer to standards
k, y refer to unknowns

calculate concentrations in mg/mL

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