

## Redox Chemistry

In the first week of this experiment you will synthesize a sample of the compound copper(II) glycinate monohydrate, which you will analyze the next week. You will also use standard reduction potentials to predict what happens in a series of redox reactions. You will perform these chemical reactions and compare your experimental results to your predicted results.

In the second week of this experiment you will standardize (determine the concentration of) a solution of sodium thiosulfate. You will then use that sodium thiosulfate solution to titrate your copper (II) glycinate you synthesized the previous week and find the mass percent of copper in copper (II) glycinate monohydrate.

### EQUIPMENT

#### **Week 1**

For part A you will need a 250 mL beaker, a 150 mL beaker, a buchner funnel with vacuum flask and hose, a piece of filter paper for the buchner funnel, 2 stirring rods, an ice bath, label tape, beaker tongs, and a small wash bottle for acetone. For part B you will need a well plate.

#### **Week 2**

For part C you will need a 500 mL Erlenmeyer flask with a rubber stopper, 2 weigh boats, three 250 mL Erlenmeyer flasks with rubber stoppers, a small beaker, a 50 mL graduated cylinder, and a 50 mL burette.

### CHEMICALS

#### **Week 1**

For part A you will need about 1.6 grams of copper(II) acetate monohydrate, about 1.3 grams of glycine, about 20 mL of 1-propanol, and a small wash bottle with acetone. For part B you will need the chemicals listed in the reaction table below.

#### **Week 2**

For part C you will need enough sodium thiosulfate pentahydrate to make 300 mL of a 0.11 M solution, about 1.8 grams of potassium ferricyanide, about 8 grams of potassium iodide, about 3 mL of 6 M hydrochloric acid, 36 mL of 1.0 M zinc sulfate,

105 mL of 0.05 M H<sub>2</sub>SO<sub>4</sub>, and 12 mL of 0.4% starch solution. You will also need the copper(II) glycinate you synthesized during week 1.

### **WASTE DISPOSAL**

Any copper containing compound should go in the copper waste container. Any acetone or 1-propanol (that does not have copper in it) should go in the organic waste container.

### **SAFETY**

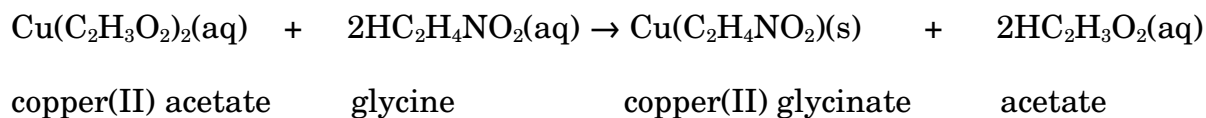
Wear your goggles the entire time. Acetone, 1-propanol, and their fumes are flammable, do not get them near any flames or sparks. Hydrochloric acid is corrosive, do not get any on you. If you do, wash the affected area with soap and water.

### **PROCEDURE**

#### **WEEK 1**

##### **Part A: Synthesis of Copper(II) Glycinate**

The chemical equation describing this reaction is:



Weigh about 1.6 grams of copper(II) acetate monohydrate into tared 250 mL beaker. Record the mass to three places past the decimal in your data table. Add about 20 mL of D.I. water to the beaker.

Weigh about 1.3 grams of glycine into a tared 150 mL beaker and record the mass to three places past the decimal in your data table. Add about 15 mL of D.I. water to the beaker.

Weigh a piece of filter paper and record the mass to three places past the decimal in your data table.

Label a watch glass with tape and a pen as "Copper(II) Glycinate" and then weigh the watch glass. Record the mass to three places past the decimal in your data table.

Place both beakers on a hot place and heat to boiling. Stir each beaker with a different stirring rod while heating to dissolve both compounds.

Once both compounds have both dissolved and are boiling or close to it carefully remove both beakers from the hot plate with **beaker tongs**.

Add the glycine solution to the copper(II) acetate solution and stir. Allow the solution to cool for a few minutes then place the beaker in an ice bath.

After crystals start to form add 20 mL of 1-propanol and continue to stir. Once the crystals have fully formed continue to cool in the ice bath for about 5 more minutes.

Place the piece of filter you weighed earlier into the buchner funnel, wet it with D.I. water and turn on the vacuum. Transfer your product into the buchner funnel, washing the beaker with acetone to make sure all of your product has been transferred onto the filter paper.

Continue pulling air through your product with the vacuum for a few more minutes. Transfer all of the product along with the filter paper onto the pre-weighed watch glass.

Place the watch glass with your product and the filter in your locker until next week.

### **Part B: Predicting Spontaneity From Reduction Potentials**

For each of the reactions listed in the following table write down the net ionic equation and the two half reactions based on the net ionic equation. Write these in your calculations section and include the standard reduction potential for each half reaction (you can look these up in your text book, the CRC Handbook of Chemistry and Physics, or online (be careful there).

Calculate the standard reduction potential for each reaction and write these next to the net ionic equation for each in your calculations section. Note in your lab report which reactions are predicted to be spontaneous and which are predicted to be non-spontaneous. The reactions with a positive potentials are predicted to be positive because:

$$\Delta G^{\circ} = -nFE^{\circ}$$

where  $n$  is the number of electrons transferred,  $F$  is called Faraday's constant and is the charge on 1 mol of electrons:  $1 F = 96,485$  coulombs/mol  $e^-$ .  $E^\circ$  is the standard potential for the reaction. Because  $\Delta G^\circ$  must be negative for a reaction to be spontaneous (under standard conditions)  $E^\circ$  must be positive.

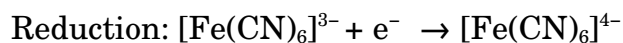
Using approximately 2 mL of each reactant perform every reaction in the table and note, in your data table, your observations for each reaction and whether or not a reaction occurred.

<u>REACTIONS</u>
1. $\text{Mg(s)} + \text{ZnSO}_4(\text{aq}) \rightarrow \text{Zn(s)} + \text{MgSO}_4(\text{aq})$
2. $\text{Cu(s)} + \text{ZnSO}_4(\text{aq}) \rightarrow \text{Zn(s)} + \text{CuSO}_4(\text{aq})$
3. $\text{Zn(s)} + \text{CuSO}_4(\text{aq}) \rightarrow \text{Cu(s)} + \text{ZnSO}_4(\text{aq})$
4. $\text{Zn(s)} + 2 \text{HCl(aq)} \rightarrow \text{ZnCl}_2(\text{aq}) + \text{H}_2(\text{g})$
5. $\text{Cu(s)} + 2\text{HCl(aq)} \rightarrow \text{CuCl}_2(\text{aq}) + \text{H}_2(\text{g})$
6. $4\text{KI(aq)} + 2\text{CuSO}_4(\text{aq}) \rightarrow 2\text{CuI(s)} + \text{I}_2(\text{aq}) + 2\text{K}_2\text{SO}_4(\text{aq})$
7. $2\text{FeCl}_3(\text{aq}) + 6\text{KI(aq)} \rightarrow 2\text{FeI}_2(\text{aq}) + \text{I}_2(\text{aq}) + 6\text{KCl(aq)}$
8. $2\text{FeCl}_3(\text{aq}) + 6\text{KBr(aq)} \rightarrow 2\text{FeBr}_2(\text{aq}) + \text{I}_2(\text{aq}) + 6\text{KBr(aq)}$
9. $\text{I}_2(\text{aq}) + \text{Na}_2\text{S}_2\text{O}_3(\text{aq}) \rightarrow 2\text{NaI(aq)} + \text{Na}_2\text{S}_4\text{O}_6(\text{aq})$

## WEEK 2

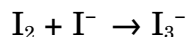
### **Part C: Standardization of A Sodium Thiosulfate Solution**

In this part of the experiment the ferricyanide ion (from potassium ferricyanide) is reduced to the ferrocyanide ion:

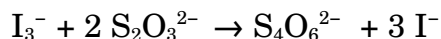


and the iodide ion is oxidized to iodine, which then complexes with more of the iodide ion to form triiodide:

Oxidation:  $2 \text{I}^- \rightarrow \text{I}_2 + 2 \text{e}^-$

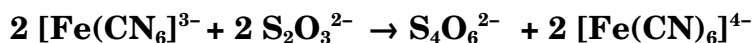


This triiodide is titrated with the sodium thiosulfate solution that you are standardizing:



Because the ferrocyanide ion makes it hard to see the endpoint of this titration it is removed as it forms by adding  $\text{ZnSO}_4$  to the solution. Starch make it easier to see the endpoint.

The overall net ionic equation describing this reaction is:



Before coming to lab calculate the mass of sodium thiosulfate pentahydrate ( $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ ) needed to make 300 mL of a 0.11 M solution of sodium thiosulfate. Show this calculation in your calculations section.

Weigh, into a tared weigh boat, approximately this amount ( no need to get the exact mass).

Add the sodium thiosulfate pentahydrate to about 300 mL of D.I. water in a 500 mL Erlenmeyer flask and swirl to dissolve. Put a stopper in the flask and set it aside.

Weigh about 0.6 grams of  $\text{K}_3\text{Fe}(\text{CN})_6$  into a tared 250 mL Erlenmeyer flask. Record the mass of  $\text{K}_3\text{Fe}(\text{CN})_6$  in your data table to 3 places past the decimal. Add 25 mL of D.I. water with a graduated cylinder. Label this as flask #1.

Repeat for two more 250 mL Erlenmeyer flasks labeling them as #2 and #3 and stopper them.

In a small beaker dissolve about 1.5 grams of KI in 10 mL of D.I. water and add 1 mL of 6 M HCl. Add this solution to the  $\text{K}_3\text{Fe}(\text{CN})_6$  solution in flask #1 and put a rubber stopper in the Erlenmeyer flask and put it in your locker drawer. Close the drawer (it needs a few minutes in private to react).

While you are waiting clean and rinse a 50 mL burette with the sodium thiosulfate solution. Fill the burette with the sodium thiosulfate solution, remove any air

bubbles from the tip and record the initial volume of sodium thiosulfate solution in your data table.

After flask #1 has been in your locker drawer for 5 minutes or so take it out , add 12 mL of 1.0 M zinc sulfate solution to it, and swirl.

In the small beaker dissolve another 1.5 grams of KI in 10 mL of D.I. water and add 1 mL of 6 M HCl. Add this solution to the  $K_3Fe(CN)_6$  solution in flask #2 and put a rubber stopper in the Erlenmeyer flask and put it in your locker drawer. Close the drawer.

Now titrate the solution in flask #1 with the sodium thiosulfate solution in the burette until the iodine color starts to fade. Then add about 2 mL of 0.4% starch solution and continue titrating to the endpoint. The endpoint is when the color changes from gray to white. Record the final volume of the sodium thiosulfate solution in the burette in your data table.

Take flask #2 out of your locker drawer, add 12 mL of 1.0 M zinc sulfate solution to it, and swirl.

In the small beaker dissolve another 1.5 grams of KI in 10 mL of D.I. water and add 1 mL of 6 M HCl. Add this solution to the  $K_3Fe(CN)_6$  solution in flask #3 and put a rubber stopper in the Erlenmeyer flask and put it in your locker drawer. Close the drawer.

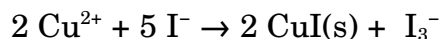
Record the initial volume of your burette and titrate the solution in flask #2 with the sodium thiosulfate solution in the burette until the iodine color starts to fade. Then add about 2 mL of 0.4% starch solution and continue titrating to the endpoint. The endpoint is when the color changes from gray to white. Record the final volume of the sodium thiosulfate solution in the burette in your data table.

Take flask #3 out of your locker drawer, add 12 mL of 1.0 M zinc sulfate solution to it, and swirl.

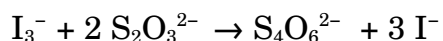
Record the initial volume of your burette and titrate the solution in flask #3 with the sodium thiosulfate solution in the burette until the iodine color starts to fade. Then add about 2 mL of 0.4% starch solution and continue titrating to the endpoint. The endpoint is when the color changes from gray to white. Record the final volume of the sodium thiosulfate solution in the burette in your data table.

## Part D: Titration of Copper (II) Glycinate

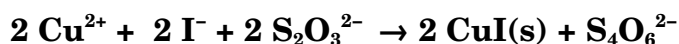
The reaction that you are using to analyze your product from last week involves a redox reaction between the copper (II) ion in your copper (II) glycinate and iodide:



The triiodide ion then reacts with the thiosulfate ion as in the standardization above:



The overall net ionic equation is:



Weigh about 0.3 grams of your copper (II) glycinate product from last week into a tared weigh boat. Record the mass to three places past the decimal in your data table.

Transfer all of the copper (II) glycinate to a clean 250 mL Erlenmeyer flask (clean one of the ones you used for the standardization of sodium thiosulfate). Use D.I. water from your wash bottle to get all of the copper (II) glycinate into the Erlenmeyer flask.

Add about 35 mL of 0.05 M  $\text{H}_2\text{SO}_4$  to the Erlenmeyer flask. Add about 1.0 gram of KI to the flask and swirl to mix.

Titrate the solution with your standardized sodium thiosulfate solution until the triiodide color starts to fade. Add about 2 mL of 0.4% starch solution and continue titrating to the endpoint (the color of the solution changes from gray to white).

Make sure to record the initial and final volumes in your data table.

Repeat for 2 more samples of your copper (II) glycinate.

## CALCULATIONS

### **Part A:**

None

### **Part B:**

Write the oxidation half-reaction and the reduction half-reaction for each of the reactions listed.

Calculate the standard potential of each reaction listed.

### **Part C:**

Calculate the mass of sodium thiosulfate pentahydrate ( $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ ) needed to make 300 mL of a 0.11 M solution of sodium thiosulfate.

Then calculate:

- 1.) The moles of  $[\text{Fe}(\text{CN}_6)]^{3-}$  in each of the 250 mL Erlenmeyer flasks.
- 2.) The moles of  $\text{S}_2\text{O}_3^{2-}$  added at the endpoint for each titration.
- 3.) The  $[\text{S}_2\text{O}_3^{2-}]$  from each of the three titrations.
- 4.) The average  $[\text{S}_2\text{O}_3^{2-}]$  for the three titrations.

### **Part D:**

Calculate:

- 1.) The theoretical mass percent copper in copper (II) glycinate –  $\text{Cu}(\text{C}_2\text{H}_4\text{NO}_2)_2$
- 2.) The moles of  $\text{S}_2\text{O}_3^{2-}$  added at the endpoint for each titration.
- 3.) The moles of  $\text{Cu}^{2+}$  in the copper (II) glycinate for each titration.
- 4.) The mass of  $\text{Cu}^{2+}$  in the copper (II) glycinate for each titration.
- 5.) The mass percent of copper in each sample of copper (II) glycinate.
- 6.) The average mass percent copper in your copper (II) glycinate.
- 7.) The percent error.



## **CONCLUSION**

**Part A:** None

**Part B:** Report which reactions occurred and which did not. Compare this list to your predictions based on the standard potential calculated for each reaction. Explain any discrepancies.

**Part C:** Report the average  $[\text{S}_2\text{O}_3^{2-}]$  for the three titrations.

**Part D:** Report the theoretical mass percent copper in copper (II) glycinate monohydrate.

Report your average mass percent copper in your sample of copper (II) glycinate monohydrate.