# Copper (II) Glycinate Titration

In 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 last week and find the mass percent of copper in copper (II) glycinate monohydrate.

## EQUIPMENT

For part A you will need a 500 mL Erlenmeyer flask with a rubber stopper, 5 weigh boats, three 250 mL Erlenmeyer flasks with rubber stoppers, a small beaker, a 50 mL graduated cylinder, and a 50 mL burette.

## CHEMICALS

For part A 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 $H_2SO_4$, and 12 mL of 0.4% starch solution.

## WASTE DISPOSAL

Any copper containing compound should go in the copper waste container.

## SAFETY

Wear your goggles the entire time. Be careful not to get hydrochloric acid on you.

## PROCEDURE

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

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

Reduction: $[Fe(CN_6)]^{3-} + e^- \rightarrow [Fe(CN_6)]^{4-}$

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

Oxidation: $2\ I^- \rightarrow I_2 + 2\ e^-$

$I_2 + I^- \rightarrow I_3^-$

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

$I_3^- + 2\ S_2O_3^{2-} \rightarrow S_4O_6^{2-} + 3\ I^-$

Because the ferrocyanide ion makes it hard to see the endpoint of this titration it is removed as it forms by adding $ZnSO_4$ to the solution. Starch make it easier to see the endpoint.

The overall net ionic equation describing this reaction is:

$$\mathbf{2\ [Fe(CN_6)]^{3-} + 2\ S_2O_3^{2-} \rightarrow S_4O_6^{2-} + 2\ [Fe(CN_6)]^{4-}}$$

Before coming to lab calculate the mass of sodium thiosulfate pentahydrate ($Na_2S_2O_3 \cdot 5H_2O$) 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 $K_3Fe(CN)_6$ into a tared 250 mL Erlenmeyer flask. Record the mass of $K_3Fe(CN)_6$ 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 $K_3Fe(CN)_6$ 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 B: 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:

$2\ Cu^{2+} + 5\ I^- \rightarrow 2\ CuI(s) + I_3^-$

The triodide ion then reacts with the thiosulfate ion as in the standardizatin above:

$I_3^- + 2\ S_2O_3^{2-} \rightarrow S_4O_6^{2-} + 3\ I^-$

The overall net ionic equation is:

$$\mathbf{2\ Cu^{2+} + 2\ I^- + 2\ S_2O_3^{2-} \rightarrow 2\ CuI(s) + S_4O_6^{2-}}$$

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 $H_2SO_4$ 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.

**<u>CALCULATIONS</u>**

Part A:

Before coming to lab calculate the mass of sodium thiosulfate pentahydrate ($Na_2S_2O_3 \cdot 5H_2O$) needed to make 300 mL of a 0.11 M solution of sodium thiosulfate.

Then calculate:

1.) The moles of $[Fe(CN_6)]^{3-}$ in each of the 250 mL Erlenmeyer flasks.

2.) The moles of $S_2O_3^{2-}$ added at the endpoint for each titration.

3.) The $[S_2O_3^{2-}]$ from each of the three titrations.

4.) The average $[S_2O_3^{2-}]$ for the three titrations.

Part B:
Calculate:
1.) The theoretical mass percent copper in copper (II) glycinate monohydrate – $Cu(C_2H_4NO_2)_2 \cdot H_2O$

2.) The moles of $S_2O_3^{2-}$ added at the endpoint for each titration.

3.) The moles of $Cu^{2+}$ in the copper (II) glycinate for each titration.

4.) The mass of $Cu^{2+}$ 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:

Report the average $[S_2O_3^{2-}]$ for the three titrations.

Discuss at least 2 possible sources of error.

Part B:

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.

Report your percent error.

Discuss at least 2 possible sources of error.