CH-412-LA Experiment 3

Oxidative and reductive properties of H_2O_2 and H_2S; precipitation and dissolution of sulfide compounds.

Max Shi, Brynn Froehlich, Erika McCarthy, Emily McGuire

I pledge my honor that I have abided by the Stevens Honor System.

Procedure

Oxidative and Reductive Activity of H2O2

1. Mix equal parts 0.2M $Pb(NO_3)_2$ and H_2S solution. After removing the supernatant, add 30% H_2O_2 solution.
2. Add several drops of 0.01M $KMnO_4$ to a test tube, then add a small amount of 0.1M H_2SO_4. Add 6% H_2O_2 solution to the test tube and observe what happens.

Metal Sulfide

3. Prepare 3 test tubes with 1.0mL of H_2S solution (0.2M), then add 0.5mL of 0.2 M $CuSO_4$, $Hg(NO_3)_2$, and $SnCl_2$ to respective tubes.
4. Centrifuge the tubes, collect the precipitates, and observe their colors.
5. Add a small amount of 6M HCl to the CuS to see if the precipitate dissolves. After centrifuging and removing the supernatant, add a small amount of HNO_3 to the tube and the precipitate dissolving.
6. Add a small amount of sulfur powder to a test tube, followed by a small amount of 0.5M Na_2S.
7. Heat the sample in a 60°C water bath and observe the color change. Add this solution into the SnS_2 precipitate test tube and observe the dissolution of the precipitate. Add 2.0 M HCl into the transparent solution and observe what happens.

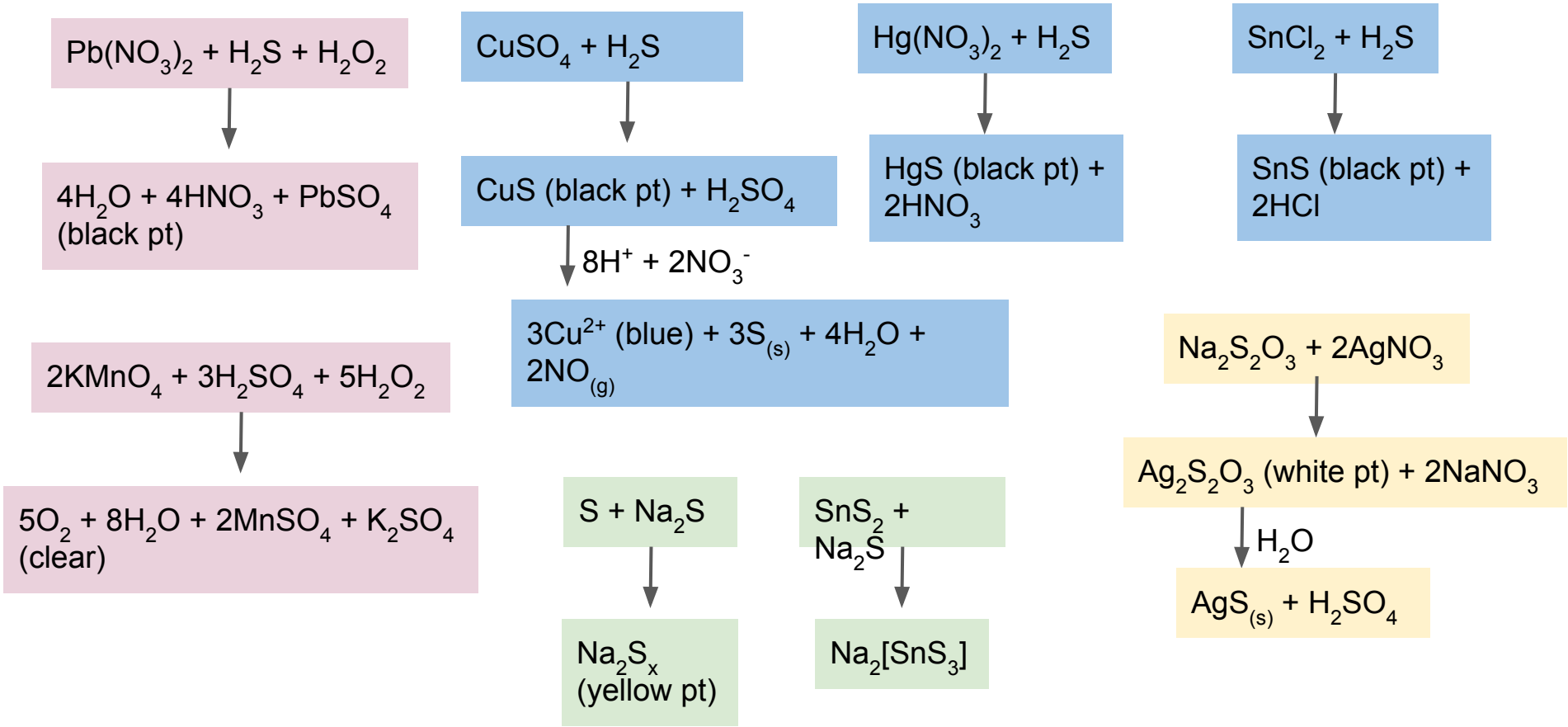
Procedure (Continued)

Properties of Na2S2O3

7. Add several drops of 0.5 M $Na_2S_2O_3$ solution into a test tube containing 0.5 mL of 0.1 M $AgNO_3$. Keep adding drops until 5.0 mL of $Na_2S_2O_3$ has been added. Observe the reaction.

8. Separately, add 5.0 mL of 0.5 M $Na_2S_2O_3$ to a test tube. Add 0.5 mL of 0.1 M $AgNO_3$ solution. Observe the reaction and compare to the previous reaction.

Flow Chart

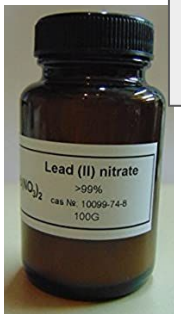

$Pb(NO_3)_2 + H_2S + H_2O_2$
$4H_2O + 4HNO_3 + PbSO_4$ (black pt)
$CuSO_4 + H_2S$
CuS (black pt) + H_2SO_4
$8H^+ + 2NO_3^-$
$3Cu^{2+}$ (blue) + $3S_{(s)}$ + $4H_2O$ + $2NO_{(g)}$
$Hg(NO_3)_2 + H_2S$
HgS (black pt) + $2HNO_3$
$SnCl_2 + H_2S$
SnS (black pt) + $2HCl$
$2KMnO_4 + 3H_2SO_4 + 5H_2O_2$
$5O_2 + 8H_2O + 2MnSO_4 + K_2SO_4$ (clear)
$S + Na_2S$
Na_2S_x (yellow pt)
SnS_2 + Na_2S
$Na_2[SnS_3]$
$Na_2S_2O_3 + 2AgNO_3$
$Ag_2S_2O_3$ (white pt) + $2NaNO_3$
H_2O
$AgS_{(s)} + H_2SO_4$

Part 1

$$Pb(NO_3)_2 + H_2S + H_2O_2 \rightarrow 4H_2O + 4HNO_3 + PbSO_4$$

Oxidative and Reductive activity of H_2O_2

1. Mix equal volume of 0.2 M $Pb(NO_3)_2$ and H_2S solution.
2. Remove supernatant and add 30% H_2O_2 solution.



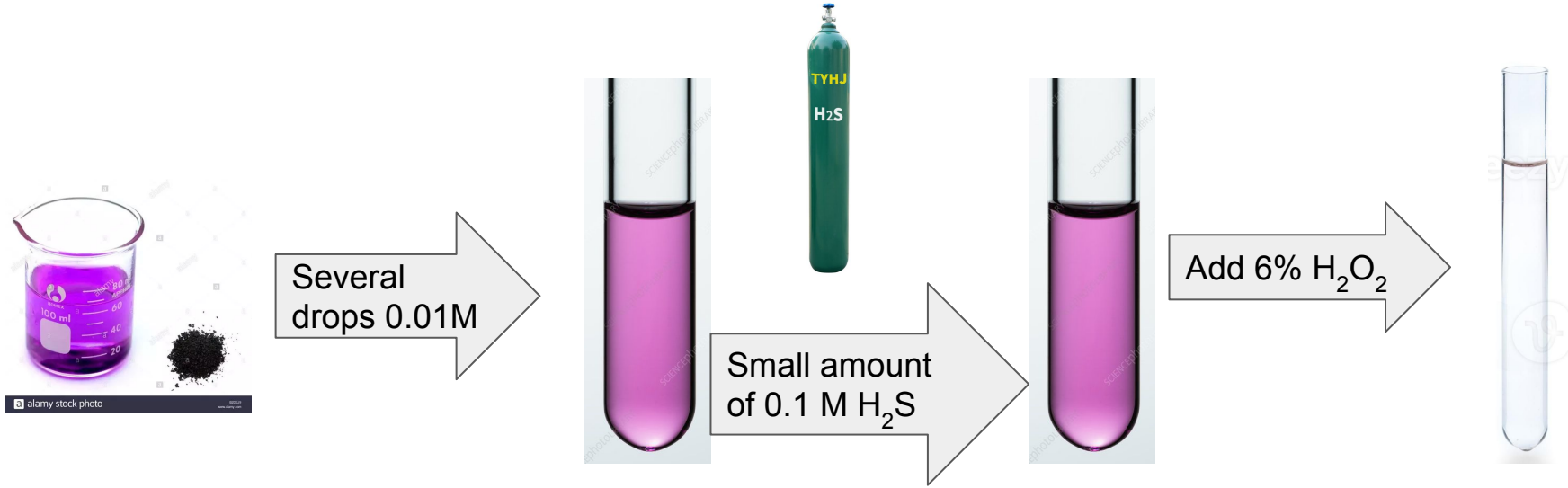
Prepared 0.2 M solutions of each

Equal volume

Remove supernatant, add 30% H_2O_2

Oxidative and reductive activity of H_2O_2 (cont.)

1. Add several drops of 0.01M $KMnO_4$ in a test tube, followed by small amount of 0.1 M H_2S.
2. Add 6% H_2O_2 solution in the test tube and observe.



$$2KMnO_4 + 3H_2SO_4 + 5H_2O_2 \longrightarrow 5O_2 + 8H_2O + 2MnSO_4 + K_2SO_4$$

Part 2

Metal sulfides

$CuSO_4 + H_2S \rightarrow CuS + H_2SO_4$

$Hg(NO_3)_2 + H_2S \rightarrow HgS + 2HNO_3$

$SnCl_2 + H_2S \rightarrow SnS + 2HCl$

1. Respectively add 0.5 mL 0.2M $CuSO_4$, $Hg(NO_3)_2$, and $SnCl_2$ solution into three test tubes containing 1.0 mL H_2S solution (0.2 M).
2. Centrifuge, collect precipitations, and observe color difference.

1.0 mL 0.2 M each

0.5 mL 0.2M $CuSO_4$

0.5 mL 0.2M $Hg(NO_3)_2$

0.5 mL 0.2M $SnCl_2$

Collect precipitates

Copper (II) sulfide

Lead (II) sulfide

Tin (II) sulfide

$$CuS + H_2SO_4$$

$$\downarrow 8H^+ + 2NO_3^-$$

$$3Cu^{2+} + 3S_{(s)} + 4H_2O + 2NO_{(g)}$$

Metal sulfides (cont.)

3. Add small amount of 6M HCl to CuS test tube and see if precipitation will dissolve.
4. Add small amount of HNO_3 into the test tube and observe precipitation dissolution.

Copper (II) sulfide

Small amount 6M HCl

Small amount HNO_3

$$S + Na_2S \rightarrow Na_2S_x$$

Metal sulfides (cont.)

1. Add small amount of sulfur powder into a test tube, followed by small amount of 0.5 M Na_2S. Heat sample in 60℃ water bath and observe color change.

Sulfur powder

Small amount sodium sulfide

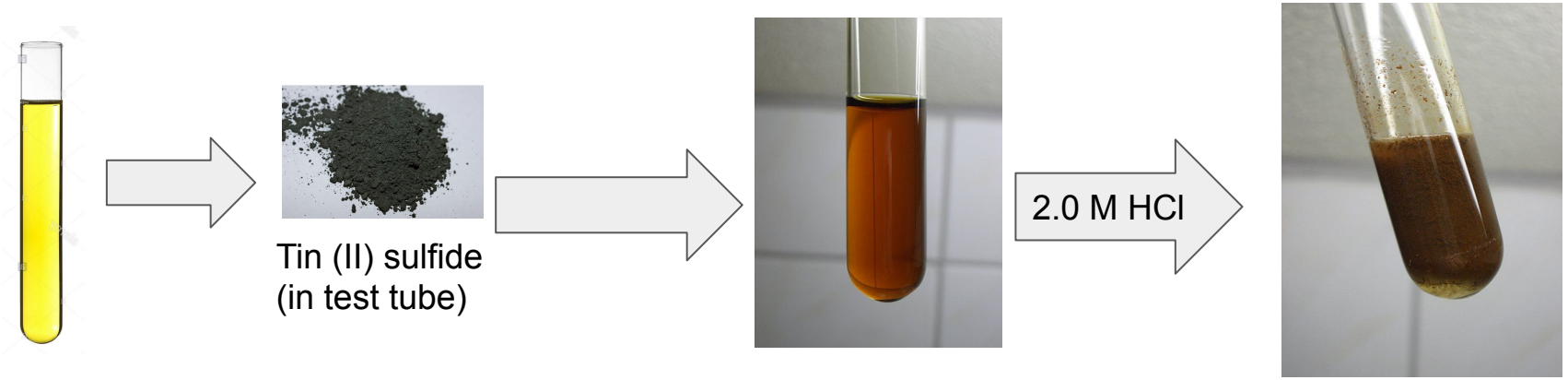
60℃ water bath

SnS_2 +
Na_2S

$Na_2[SnS_3]$

Metal Sulfides (cont.)

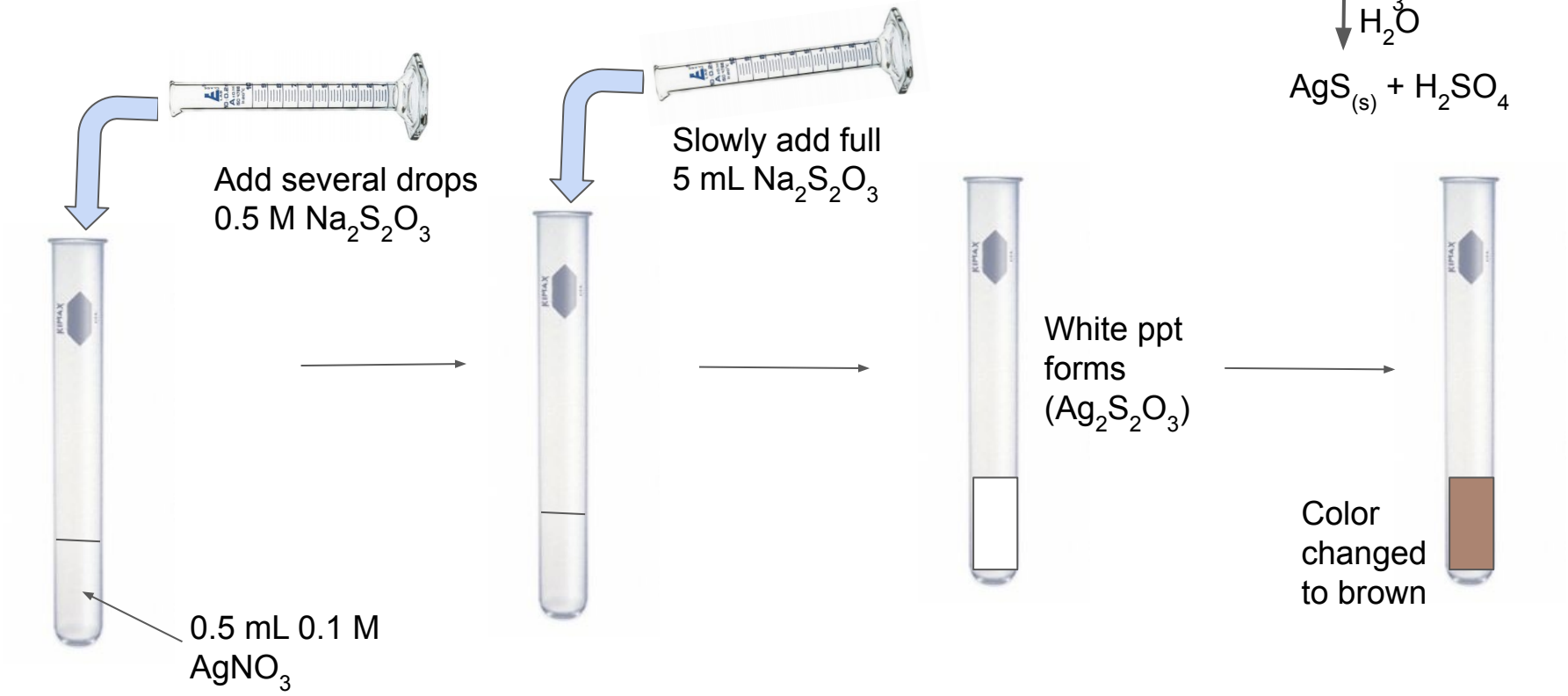
2. Add this solution into SnS_2 precipitation test tube and observe precipitation dissolution. Add 2.0 M HCl into above transparent solution and observe results.



Part 3

Properties of $Na_2S_2O_3$

$$Na_2S_2O_3 + 2AgNO_3 \rightarrow Ag_2S_2O_3 + 2NaNO_3 \xrightarrow{H_2O} AgS_{(s)} + H_2SO_4$$



Properties of $Na_2S_2O_3$

New test tube

Add 0.5 mL $AgNO_3$

5 mL $Na_2S_2O_3$

White ppt initially forms ($Ag_2S_2O_3$)

Becomes light yellow-brown ppt

Conclusion

Accomplished: While completing this lab we were able to illustrate the oxidative and reductive properties of H_2O_2 and H_2S. We demonstrated the formation of multi sulfurs compounds. The reagents used in this experiment were H_2O_2, $Pb(NO_3)_2$, H_2S, $KMnO_4$, H_2SO_4, $CuSO_4$, $Hg(NO_3)_2$, $SnCl_2$, HCl, CuS, HNO_3, Na_2S, SnS_2, $Na_2S_2O_3$, $AgNO_3$. The techniques used were centrifuging, mixing, and observation.

What was learned: We learned that when $Pb(NO_3)_2$ and H_2S are added together they form a black precipitate. When H_2O_2 is added to this, the precipitate turns white. When $KMnO_4$ and H_2SO_4 are mixed, the solution is purple. When H_2O_2 is added to this, the solution turns clear. $CuSO_4$ starts as blue and when it is added to H_2S, it turns brown with a dark precipitate. $SnCl_2$ is white/milky and when it is added to H_2S, it turns brown with precipitate. CuS and HCl together don't have a reaction. When HNO_3 is added to this, the solution turns yellow. Sulfur powder with Na_2S in heat caused a color change from clear solution with white powder on top to a yellow solution with yellow precipitate on top. When this is added to SnS_2, the precipitate dissolves and the solution turns green. The addition of HCl makes the solution turn yellow with an orange precipitate. $Na_2S_2O_3$ added to $AgNO_3$ turns from clear to pale yellow with a white precipitate with the addition of addition $Na_2S_2O_3$. With more, the precipitate turns black and then dissolves. $AgNO_3$ added to NaS_2O_3 changes from a yellow precipitate to a brown precipitate.

Any issues during the experiment: Since the experiment was done virtually without calculations there were no issues, however a typical issue that may have occurred in the lab could have been heating and mixing for correct amount of time. If either of these were to be executed poorly then the reaction may not fully develop the precipitates and color changes meant to take place.

Future recommendations/practical application: Understanding the importance of oxidation/reduction reactions is essential especially when it comes to applying this knowledge to other research. Hydrogen peroxide is one of the most powerful oxidizers, but it can be used as a reducing agent as well.

Postlab Questions

1. **What is the most advantage of using H2O2 as an oxidant?**

   H2O2 is advantageous to use as an oxidizing reagent due to its “pureness” because when it breaks down it forms oxygen and water.

2. **If H2S or Na2S solution is placed for a long time, what may happen to the solution?**

   The concentration will decrease as H2S or Na2S molecules bubble out of the solution.