

## Lab #4: Gravimetric and Solution Stoichiometry

### Special Background Information

In Part A of this lab, we will be studying the reaction between KOH and CuSO<sub>4</sub> solution. This reaction will yield a precipitate that has a tendency to absorb a bit of water (i.e. hydrates). In order to produce a precipitate that can dry quickly after filtration, a secondary reaction needs to take place where the initial product turns to a metal oxide and water.

### Objectives:

1. To accurately determine the mass of the precipitate in a precipitation reaction.
2. To accurately determine the concentration of a solution using titration.

### Pre-lab Exercise:

1. For the initial reaction between CuSO<sub>4(aq)</sub> and KOH<sub>(aq)</sub>, write the balanced molecular equation, complete ionic equation, and the net-ionic equation.
2. Use the precipitate determined in the last question as the reactant in the secondary reaction, write a balanced decomposition equation where the product is a metal oxide and water.
3. Write the balanced molecular equation for the neutralization of H<sub>2</sub>SO<sub>4(aq)</sub> with NaOH<sub>(aq)</sub>.

### Materials:

Ring Stands	One 150 mL Glass Beaker	CuSO <sub>4(aq)</sub> (0.375 M) – from Lab #3
Iron Ring	One 250 mL Glass Beakers	KOH <sub>(s)</sub> (1.44 g)
Funnel	Two 250 mL Plastic Beaker	H <sub>2</sub> SO <sub>4(aq)</sub> (0.00720 M) – from Lab #3
Filter Paper	Watch Glass and Masking Tape	NaOH <sub>(aq)</sub> (unknown concentration)
Scoopula & Stirring Rod	Buret	Bromothymol Blue Indicator
Hot Plate	Buret Funnel and Buret Clamp	Deionized Water
Pipet Bulb	4 Small / Medium Erlenmeyer Flasks	Na <sub>2</sub> CO <sub>3(s)</sub> (Optional)
Two 10 mL Pipets	50 mL to 100 mL Graduated Cylinder	Electronic Balance

### Procedure:

#### A. Precipitation Reaction:

1. Measure the mass of the filter paper.
2. Set up the filtration apparatus using the ring stand, ring, funnel and a 250 mL plastic beaker.
3. Fold a correct size filter paper for the funnel.
4. Place the folded filter paper into the funnel. Wet the paper with deionized water so it sticks inside the funnel.
5. Place the 150 mL glass beaker on the electronic balance and set it to zero.
6. Measure out approximately 1.44 g of KOH<sub>(s)</sub> in the 150 mL glass beaker. Record the actual mass of KOH<sub>(s)</sub> used.
7. Completely dissolve the KOH<sub>(s)</sub> with approximately 20 mL of deionized water and a stirring rod. Leave the stirring rod in the beaker.
8. Label a 250 mL plastic beaker as “Waste”.
9. Coat the inside of a 10 mL pipet at least twice with the 0.375 M of CuSO<sub>4(aq)</sub> from the volumetric flask as prepared in Lab #3. Discard coating solution in the “Waste” beaker.
10. Using the pipet, measure out 40.0 mL of 0.375 M CuSO<sub>4(aq)</sub>. Transfer it into a 150 mL glass beaker containing the dissolved KOH. Record any qualitative observations.
11. Place the glass beaker on the hot plate and turn it on to medium. Use the stirring rod and break up the precipitate and stir continuously as the solution heats up to a **lukewarm temperature**. You may add some more deionized water into the beaker if the evaporation of the filtrate causes a low volume of the mixture. (If you decide to do so, make sure you do not overfill the beaker). Do not let the solution boil over (turn down the heat if the solution bubbles too vigorously). Record any changes.
12. When the change observed in the last step has become permanent, (it should take around 5 to 10 minutes. **Note: If there is no change in the beaker after 10 minutes, ask the instructor to add a small amount of sodium carbonate solid into the beaker.**), carefully filter the mixture through the filtration apparatus using a stirring rod in small portions. Do not overfill the filter! Be sure to wash the beaker and the stirring rod thoroughly with deionized water.

- Label your name on the backside of the watch glass using a masking tape.
- Carefully take out the filter paper from the funnel. Open it up and place it on the watch glass to dry. Wait at least a whole day until it's completely dry. Measure and record the mass of the filter paper and precipitate without the watch glass.

**B. Acid and Base Titration:**

- Label a clean 250 mL glass beaker as NaOH. Obtain the unknown concentration of NaOH<sub>(aq)</sub> from your teacher.
- Coat the 10 mL pipet with the NaOH<sub>(aq)</sub> at least twice and discard the wash fluid in the 250 mL plastic beaker labeled as "Waste".
- Pipet 10 mL of NaOH<sub>(aq)</sub> to each of the three Erlenmeyer flasks.
- To each Erlenmeyer flask, add a few (three to five) drops of bromothymol blue indicator.
- Coat the buret with the 0.00720 M of H<sub>2</sub>SO<sub>4(aq)</sub> from the volumetric flask (as prepared in Lab #3) at least twice, and discard the wash fluid in the "waste" beaker.
- Set up the titration apparatus with the ring stand, buret clamp, buret and buret funnel.
- Fill the buret with the 0.00720 M of H<sub>2</sub>SO<sub>4(aq)</sub> using the buret funnel. Be sure not to pass the 0 mL mark.
- Record the starting volume of the H<sub>2</sub>SO<sub>4(aq)</sub>. Begin titration of the unknown concentration of NaOH<sub>(aq)</sub>. Swirl the Erlenmeyer flask when adding the H<sub>2</sub>SO<sub>4(aq)</sub>. The endpoint will be a green color. Record the final volume of the H<sub>2</sub>SO<sub>4(aq)</sub> added. Calculate the net volume of acid added. (If the solution becomes yellow, you have added too much H<sub>2</sub>SO<sub>4(aq)</sub>. Record the volume and the color anyway).
- Repeat Steps 7 and 8 twice with the other two Erlenmeyer flasks. Be sure to record the initial and final volume of the buret each time. Try to adjust the buret valve in such a way so H<sub>2</sub>SO<sub>4(aq)</sub> is added one drop at a time around the endpoint. You should obtain at least two consistent volume difference of H<sub>2</sub>SO<sub>4</sub> added. If you need to do a 4<sup>th</sup> trial, prepare another Erlenmeyer flask with the 10 mL NaOH as described from step 3.

**Observations:****Part A: Precipitation Reaction:**

Actual Mass of KOH used	
Mass of Dry Filter Paper	
Mass of Dry Filter Paper and Precipitate	
Observation(s) of the Initial and Final Precipitate formed	

**Part B: Acid and Base Titration:**

10.0 mL of NaOH <sub>(aq)</sub> titrated by 0.00720 mol/L of H <sub>2</sub> SO <sub>4(aq)</sub>				
	Trial 1	Trial 2	Trial 3	Trial 4
<b>Initial Volume</b>				
<b>Final Volume</b>				
<b>Volume of H<sub>2</sub>SO<sub>4</sub> added</b>				
<b>Bromothymol Blue Colour</b>				

**Analysis: (You MUST Present all your Work in your Lab Report)****Part A: Precipitation Reaction:**

- Determine the experimental mass of the final metal oxide precipitate.
- Calculate the theoretical mass of final metal oxide precipitate formed when 40.0 mL of 0.375 mol/L of CuSO<sub>4(aq)</sub> is reacted with the mass of KOH<sub>(s)</sub> used.
- (For students who added sodium carbonate in Part A:) What chemical reactions happened when sodium carbonate is added to the reaction mixture? Will that change the limiting reactant? Recalculate your answer in the last question based on this added fact.

**Part B: Acid and Base Titration:**

1. Determine the experimental concentration of  $\text{NaOH}_{(aq)}$ .

**Evaluation:****Part A: Precipitation Reaction:**

1. Calculate the % error of the precipitate and comment on the possible reasons for the errors.
2. Predict and explain what would happen to the experimental mass of the precipitate if the beaker containing reaction did not get washed out with deionized water.
3. Why is it unnecessary to calculate the concentration of  $\text{KOH}_{(aq)}$  used to find the theoretical mass of the precipitate form?

**Part B: Acid and Base Titration:**

1. **Predict and explain** specifically what would happen to the calculated  $[\text{NaOH}_{(aq)}]$  when there is/are
  - a. deionized water left in the Erlenmeyer flask when  $\text{NaOH}_{(aq)}$  is transferred.
  - b. deionized water left in the pipet when  $\text{NaOH}_{(aq)}$  is transferred to the Erlenmeyer flask.
  - c. air bubbles in the pipet when  $\text{NaOH}_{(aq)}$  is transferred to the Erlenmeyer flask.
  - d. deionized water left in the buret when  $\text{H}_2\text{SO}_4_{(aq)}$  is added.
  - e. air bubbles in the buret that got dislodged when  $\text{H}_2\text{SO}_4_{(aq)}$  is added.
2. The approximate theoretical concentration of  $\text{NaOH}_{(aq)}$  is 0.0540 M. Compare your calculated  $[\text{NaOH}_{(aq)}]$  to this theoretical concentration by determining the % error. What are the possible sources of error?

**Conclusion:**

1. Accounting for the % errors, what would you do to improve the procedures of this lab?
2. Choose one of the reactions from Part A or Part B, write a statement of understanding (phenomena, evidences, reasoning from a particle perspective, and claim) explaining what had happened.