

## Lab #3: Gravimetric and Solution Stoichiometry

### Objectives:

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

### Hypothesis / Pre-lab Exercise:

- Write the balanced molecular equation, complete ionic equation, and the net-ionic equation for the reaction between  $\text{Fe}(\text{NO}_3)_3(aq)$  and  $\text{Na}_2\text{CO}_3(aq)$ .
- Write the balanced molecular equation for the neutralization of  $\text{H}_2\text{SO}_4(aq)$  with  $\text{KOH}(aq)$

### Materials:

Ring Stand	Two 150 mL or 100 mL small Beakers	$\text{Fe}(\text{NO}_3)_3(aq)$ (0.250 M) – from Lab #2
Iron Ring	Two 250 mL large Beakers	$\text{Na}_2\text{CO}_3(s)$ (2.00 g)
Funnel	Buret	$\text{H}_2\text{SO}_4(aq)$ (0.0360 M) – from Lab #2
Filter Paper	Watch Glass and Masking Tape	$\text{KOH}(aq)$ (unknown concentration)
Scoopula	Buret Funnel and Buret Clamp	Bromothymol Blue Indicator
Stirring Rod	4 Small / Medium Erlenmeyer Flasks	Distilled Water
Volumetric Flasks	Two 10 mL Pipets	Electronic Balance

### Procedure:

#### A. Precipitation Reaction:

- Measure out approximately 2.00 g of  $\text{Na}_2\text{CO}_3(s)$  in a small beaker. Record the actual mass of  $\text{Na}_2\text{CO}_3(s)$  used.
- Completely dissolve the  $\text{Na}_2\text{CO}_3(s)$  with approximately 20 mL of water. Rinse the stirring rod with distilled water.
- Coat the inside of a 10 mL pipet at least twice with the 0.250 M of  $\text{Fe}(\text{NO}_3)_3(aq)$  from the volumetric flask as prepared in Lab #2. Discard it into a waste beaker (label a large beaker as Waste).
- Pipet out 60 mL of 0.250 M  $\text{Fe}(\text{NO}_3)_3(aq)$ . Transfer it into the beaker containing the  $\text{Na}_2\text{CO}_3$  solution. Write down any qualitative observation. Besides the precipitation and the colour of the precipitate, did you observe any gas formed?
- Set up the filtration apparatus using the ring stand, ring, funnel and a large beaker.
- Fold a correct size filter paper for the funnel and measure the mass of the filter paper.
- Place the folded filter paper into the funnel. Wet the paper with distilled water so it sticks inside the funnel.
- Carefully filter the mixture from step 4 using a stirring rod. Be sure to wash the beaker out thoroughly.
- Label your name on a 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 small beaker as KOH and obtain the solution from your teacher.
- Coat the 10 mL pipet with the  $\text{KOH}(aq)$  at least twice and discard the wash fluid in a large beaker labeled as "Waste" (from step 1 in the last section).
- Pipet 10 mL of  $\text{KOH}(aq)$  to each of the three Erlenmeyer flasks.
- To each Erlenmeyer flask, add a few drops of bromothymol blue indicator.
- Coat the buret with the 0.0360 M of  $\text{H}_2\text{SO}_4(aq)$  from the volumetric flask (as prepared in Lab #2) 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 0.0360 M of  $\text{H}_2\text{SO}_4(aq)$  using the buret funnel. Be sure not to pass the 0 mL mark.
- Record the starting volume of the  $\text{H}_2\text{SO}_4(aq)$ . Begin titration of the unknown concentration of  $\text{KOH}(aq)$ . Swirl the Erlenmeyer flask when adding the  $\text{H}_2\text{SO}_4(aq)$ . The endpoint will be a green color. Record the final volume of the  $\text{H}_2\text{SO}_4(aq)$  added. Calculate the net volume of acid added. (If the solution becomes yellow, you have added too much  $\text{H}_2\text{SO}_4(aq)$ . Record the volume and the colour anyway).
- Repeat Step 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  $\text{H}_2\text{SO}_4(aq)$  is added one drop at a time around the endpoint. Obtain at least two consistent trials (that means do a 4<sup>th</sup> trial if necessary).

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

Actual Mass of Na <sub>2</sub> CO <sub>3</sub> used	
Mass of Dry Filter Paper	
Mass of Dry Filter Paper and Precipitate	
Observation(s) of the Precipitate formed and overall reaction	

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

10.0 mL of KOH <sub>(aq)</sub> titrated by 0.0360 mol/L of H <sub>2</sub> SO <sub>4 (aq)</sub>				
	Trial 1	Trial 2	Trial 3	Trial 4 (optional)
Initial Volume				
Final Volume				
Volume of H <sub>2</sub> SO <sub>4</sub> added				
Bromothymol Blue Colour				

**Analysis:****Part A: Precipitation Reaction:**

- Determine the experimental mass of the precipitate.
- Calculate the theoretical mass of precipitate formed when 60.0 mL of 0.250 mol/L of Fe(NO<sub>3</sub>)<sub>3 (aq)</sub> is reacted with the mass of Na<sub>2</sub>CO<sub>3 (s)</sub> used.
- Calculate the concentration of the ions for the filtrate (assuming all the reactants are transferred completely into the beaker and no washing fluid is needed).

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

- Determine the experimental concentration of KOH<sub>(aq)</sub>.

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

- Calculate the % error of the precipitate and comment on the possible reasons for the errors.
- Predict and explain what would happen to the experimental mass of the precipitate if the beaker containing Fe(NO<sub>3</sub>)<sub>3 (aq)</sub> did not get washed out with distilled water.
- Why is it unnecessary to calculate the concentration of Na<sub>2</sub>CO<sub>3 (aq)</sub> used to find the theoretical mass of the precipitate form?
- Go to <http://www.chemguide.co.uk/inorganic/transition/iron.html>. Read the section “iron (III) ion and carbonate ion”. Propose another possible source of error. Design a follow-up experiment that could determine the identity of the precipitate as well determining if it is a hydrate.

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

- Predict and explain what would happen to the calculated [KOH<sub>(aq)</sub>] when there is/are
  - distilled water left in the Erlenmeyer flask when KOH<sub>(aq)</sub> is transferred.
  - distilled water left in the pipet when KOH<sub>(aq)</sub> is transferred to the Erlenmeyer flask.
  - air bubbles in the pipet when KOH<sub>(aq)</sub> is transferred to the Erlenmeyer flask.
  - distilled water left in the buret when H<sub>2</sub>SO<sub>4 (aq)</sub> is added.
  - air bubbles in the buret when H<sub>2</sub>SO<sub>4 (aq)</sub> is added.
- The approximate theoretical concentration of KOH<sub>(aq)</sub> is 0.162 M. Compare your calculated [KOH<sub>(aq)</sub>] with this theoretical concentration by determining the % error. What are the possible sources of error?

**Conclusion:**

- Accounting for the % errors, what would you do to improve the procedures of this lab?
- Summarize what you have learned from this lab.