#### **EXPERIMENT A4: PRECIPITATION REACTION AND THE LIMITING REAGENT**

### **Learning Outcomes**

Upon completion of this lab, the student will be able to:

- 1) Demonstrate the formation of a precipitate in a chemical reaction.
- 2) Distinguish between limiting reagent and excess reagent in a chemical reaction.
- 3) Measure the amount of precipitate formed in a chemical reaction and relate it to the amount of the limiting reagent.

### Introduction

Some ionic substances are only sparingly soluble in water. When the ions of such substances are combined in an aqueous medium, a solid precipitate may result. The formation of a precipitate depends on two factors: 1) the solubility of the substance (this concept will be discussed further when solubility rules are studied) and 2) the amounts of the ions present in the aqueous medium (this is based on the concept of solubility product constant which will be discussed later on in General Chemistry).

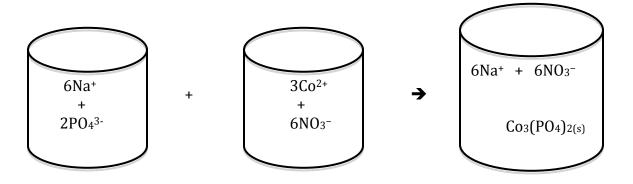
A precipitation reaction is a type of chemical reaction in which the combination of two aqueous solutions results in the formation of a precipitate. The specific reaction that will be studied in this experiment is the one between aqueous solutions of sodium phosphate and cobalt (II) nitrate. The molecular equation of the resulting chemical reaction is shown below:

 $2Na_3PO_{4(aq)} + 3Co(NO_3)_{2(aq)} \rightarrow Co_3(PO_4)_{2(s)} + 6NaNO_{3(aq)}$ 

Since sodium phosphate and cobalt (II) nitrate are both soluble ionic substances, they will dissociate into their respective ions when dissolved in water. When the two solutions are combined, the combination of the  $Co^{2+}(aq)$  and  $PO_{4^{3-}(aq)}$  results in the formation of the insoluble cobalt (II) phosphate. The net ionic equation and a pictorial representation of this process are given below. In the combined solution, the Na<sup>+</sup>(aq) and the NO<sub>3</sub>-(aq) are spectator ions.

 $2PO_{4^{3}}(aq) + 3CO^{2+}(aq) \rightarrow CO_{3}(PO_{4})_{2(s)}$ 

#### **Pictorial Representation**



The amount of precipitate formed depends the amounts of the reagents that are being combined. Based on the balanced molecular equation (see above), we can see that two moles of sodium phosphate reacts with three moles of cobalt (II) nitrate to form one mole of cobalt (II) phosphate precipitate. If the ratio of the moles of sodium phosphate and cobalt (II) nitrate is different from 2:3 then the moles of the precipitate of cobalt (II) phosphate will also be different.

For instance: when one mole of sodium phosphate is combined with three moles of cobalt (II) nitrate, then only half a mole of the precipitate of cobalt (II) phosphate will result. Since the moles of the sodium phosphate in this situation is less than the required two moles (needed to combine with the three moles of cobalt (II) nitrate), the sodium phosphate is considered to be the limiting reagent. The limiting reagent determines the amount of product that can be formed, because it is completely consumed in the reaction. In the same situation, the cobalt (II) nitrate is considered to be the excess reagent, as it will not be completely consumed at the end of the reaction.

#### **Example**

Assume that the above chemical reaction is being conducted with 0.010 M aqueous solutions of sodium phosphate and cobalt (II) nitrate. In one particular experiment, 2.00 mL of sodium phosphate is combined with 20.00 mL of cobalt (II) nitrate.

# *How can the identity of the limiting reagent and the excess reagent in this experiment be determined?*

There are several different methods that can be employed to arrive at the answer. One method is to determine the available moles of each reagent and then calculate the moles of reagent 2 needed to completely react with reagent 1. If there is enough of reagent 2 to react with reagent 1, then reagent 2 is the excess reagent and reagent 1 is the limiting reagent. On the contrary, if there is not enough of reagent 2 to react with reagent 1, then reagent 2 is the limiting reagent and reagent 1 is the excess reagent.

In this example:

Moles sodium phosphate available =  $0.010 \frac{moles}{Liter} \times 2.00 ml \times \frac{1Liter}{1000 ml} = 0.000020 moles$ 

Moles cobalt (II) nitrate available =  $0.010 \frac{moles}{Liter} \times 20.00 ml \times \frac{1Liter}{1000 ml} = 0.00020 moles$ 

Now calculate the moles of cobalt (II) nitrate needed to react with the available moles of sodium phosphate:

Moles cobalt (II) nitrate needed =  $0.000020 \text{ molesNa}_3 PO_4 \times \frac{3Co(NO_3)_2}{2Na_3 PO_4} = 0.000030 \text{ moles}$ 

The amount of cobalt (II) nitrate available is larger than the amount of cobalt (II) nitrate needed, as evidenced when the number of moles available of each are compared. This implies that the cobalt (II) nitrate is available in excess, and therefore the sodium phosphate is the limiting reagent in this case.

The main ideas from the above discussion may be summarized as follows:

- The limiting reagent is the reagent that is consumed completely in the chemical reaction.
- The amount of product formed depends on the amount of the limiting reagent present.
- The amount of product than can be formed (based on the calculation) is called the **Theoretical Yield**.

#### Sample Calculations

Consider the reaction between 10.0 mL of 0.010 M sodium phosphate and 20.0 mL of 0.010 M cobalt (II) nitrate.

#### a. What is the limiting reagent in this reaction?

Moles sodium phosphate available =  $0.010 \frac{moles}{Liter} \times 10.0ml \times \frac{1Liter}{1000ml} = 0.00010moles$ 

Moles cobalt (II) nitrate available =  $0.010 \frac{moles}{Liter} \times 20.00 ml \times \frac{1Liter}{1000 ml} = 0.00020 moles$ 

Moles cobalt (II) nitrate needed =  $0.00010 \text{ molesNa}_3 PO_4 \times \frac{3Co(NO_3)_2}{2Na_3PO_4} = 0.00015 \text{ moles}$ 

The moles of cobalt (II) nitrate available are larger than the moles of cobalt (II) nitrate needed. This implies that the cobalt (II) nitrate is available in excess, and therefore the sodium phosphate is the limiting reagent in this case.

# b. Calculate the theoretical yield, in grams, of the cobalt (II) phosphate precipitate.

The yield of the product depends on the amount of the limiting reagent.

Theoretical yield =  $0.010 \frac{moles}{Liter} \times 10.0ml \times \frac{1Liter}{1000ml} \times \frac{1Co_3(PO_4)_2}{2Na_3PO_4} \times 366.7 \frac{grams}{mole} = 0.0183 grams$ 

# c. If the actual yield of cobalt (II) phosphate is 0.0175 grams, what is the percent yield of this reaction?

Percent Yield =  $\frac{Actual Yield}{Theoritical Yield} \times 100 = \frac{0.0175g}{0.0183g} \times 100 = 95.6\%$ 

### **Experimental Design**

Aqueous solutions of 0.095 M sodium phosphate and 0.100 M cobalt (II) nitrate will be provided. Different volumes of sodium phosphate will be combined with a fixed volume of cobalt (II) nitrate. In the first part of the experiment, a qualitative assessment of the chemical reaction between the two reagents will be done. In the second part of the experiment, the precipitate of cobalt (II) phosphate will be filtered, dried, and weighed. A plot of the mass of the sodium phosphate vs. cobalt (II) phosphate will be used to illustrate the effect of the limiting reagent on the amount of product formed.

### **Reagents and Supplies**

0.095 M sodium phosphate, 0.100 M cobalt (II) nitrate, solid sodium phosphate and solid cobalt (II) nitrate

(See posted Material Safety Data Sheets)

Filter paper (12.5 cm diameter), 25-mL burettes (2), funnel

### Procedure

# PART 1: QUALITATIVE PROPERTIES OF COBALT (II) NITRATE AND SODIUM PHOSPHATE INTERACTIONS

Record detailed observations of the chemical process in each of the following situations:

- 1. Dissolve 1.0 g of solid  $Co(NO_3)_2$  in 20.0 mL of deionized water. Label this solution as A. Describe the appearance of the solution.
- 2. Dissolve about 1.0 g of solid Na<sub>3</sub>PO<sub>4</sub> in 20.0 mL of deionized water. Label this solution as B. Describe the appearance of the solution.
- 3. Combine about half of solution A and half of solution B in a new beaker. Label this beaker as C. Describe the appearance of the mixture in beaker C.
- 4. Assuming that the reaction involves the coming together of dissolved ions, what are the possible identities of the solid formed in the mixture in C? Write a balanced chemical equation for this reaction. Refer to the solubility rules to correctly indicate the phase of the reactants and products.
- 5. Separate the mixture in C by filtration. The liquid that passes through the filter is called the supernatant. Collect this liquid in a beaker labeled D and predict all of the possible chemical species, which might be present in the liquid.
- 6. Divide the supernatant liquid D in half and test each half with the remaining Co(NO<sub>3</sub>)<sub>2</sub> and Na<sub>3</sub>PO<sub>4</sub> solutions from solutions A and B. Describe the results.
- 7. What conclusions can be drawn from these data concerning the chemicals present in the supernatant in beaker D? Is one reactant in excess? (How might changing the original amounts of the reactants affect the composition of the supernatant?)
- 8. Draw a picture of the mixture of Co(NO<sub>3</sub>)<sub>2</sub> and Na<sub>3</sub>PO<sub>4</sub> before the solution was filtered. The drawing should represent what occurs on a molecular level.

# PART 2: QUANTITATIVE PROPERTIES OF COBALT (II) NITRATE AND SODIUM PHOSPHATE INTERACTIONS

- 1. Obtain two 25-mL burettes from the stockroom and clamp the burettes to a burette stand.
- 2. Label one burette as 0.095 M sodium phosphate and the second burette as 0.100M cobalt (II) nitrate.
- 3. Wash the burettes with deionized water and condition the burettes with the respective reagents and fill each burette with the appropriate reagent.
- 4. Record the "Initial Burette Reading" for both burettes.

<u>NOTE: The instructor will assign three trials to each group. Follow the procedure</u> <u>below (outlined for Trial 1) for each of the assigned trials.</u>

Trial	Volume of 0.100 M Na <sub>3</sub> PO <sub>4</sub> (mL)	Volume of 0.100 M Co(NO <sub>3</sub> ) <sub>2</sub> (mL)
1	2.0	20.0
2	4.0	20.0
3	8.0	20.0
4	10.0	20.0
5	12.0	20.0
6	16.0	20.0
7	18.0	20.0
8	20.0	20.0
9	22.0	20.0
10	24.0	20.0

- 5. Add approximately 20.0 mL of cobalt (II) nitrate from the burette into a 125-mL Erlenmeyer flask. Record the final burette reading.
- 6. Add approximately 2.0 mL of sodium phosphate into the same 125-mL Erlenmeyer flask (containing cobalt (II) nitrate) from step 5.
- 7. Mix the reagents by swirling the flask gently and allow the reagents to react for exactly 10-minutes. During the waiting period, assemble the filter flask and funnel.
- 8. Obtain the mass of an empty filter paper. Label this paper as "Trial 1" using a pencil. Fold the filter paper as instructed and place it in the funnel.
- 9. Once the reaction in step 7 is complete, pour the resulting mixture onto the filter paper. Allow the mixture to filter under gravity. This will take 20-30 minutes.

- 10. Rinse the precipitate with about 3-4 mL of cold water.
- 11. Once all the precipitate is collected on the filter paper, the instructor will collect the precipitate and dry the precipitate in a pre-heated oven for approximately 30 minutes.
- 12. Cool the precipitate to room temperature and obtain the mass of the filter paper containing the dry precipitate.
- 13. Repeat steps 5-11 with different volumes (as assigned by the instructor) of sodium phosphate as indicated in the table below.
- 14. Collect all the waste material in a large beaker and dispose the waste in an appropriate waste disposal container provided by the instructor.

## Data Table

# Part 1: Qualitative properties of cobalt (II) NITRATE AND SODIUM PHOSPHATE INTERACTIONS

Step	Experiment	Observation
1	Describe the appearance of solution A. What are the possible ions in solution A?	
2	Describe the appearance of solution B. What are the possible ions in solution B?	
3	Describe the appearance of mixture C.	
4	Identity of solid in C. Write a chemical equation for the reaction that forms the solid in solution C.	
5	After filtering solution C describe the supernatant collected in beaker D. What are the possible ions remaining in the solution D.	
6A.	Describe what happened after adding half of the supernatant D to solution A.	
6B.	Describe what happened after adding the second half of supernatant D to solution B.	
7.	Based on your results in 6A and 6B, what ions must have been present in supernatant D? What reactant must have been limiting? The Co(NO <sub>3</sub> ) <sub>2</sub> found in solution A or Na <sub>3</sub> PO <sub>4</sub> found in solution B? Explain based on the results form 6A and 6B.	

# Part 2: Quantitative properties of cobalt (II) nitrate and sodium phosphate interactions

TABLE	1

	Trial 1		Tr	Trial 2		Trial 3		Trial 4		Trial 5	
	Na <sub>3</sub> PO <sub>4</sub>	Co(NO <sub>3</sub> ) <sub>2</sub>	Na <sub>3</sub> PO <sub>4</sub>	Co(NO <sub>3</sub> ) <sub>2</sub>	Na <sub>3</sub> PO <sub>4</sub>	Co(NO <sub>3</sub> ) <sub>2</sub>	Na <sub>3</sub> PO <sub>4</sub>	Co(NO <sub>3</sub> ) <sub>2</sub>	Na <sub>3</sub> PO <sub>4</sub>	Co(NO <sub>3</sub> ) <sub>2</sub>	
Initial Burette Reading (mL) Final Burette											
Reading (mL) Volume (mL)											

### <u>Table 1 (contd.)</u>

	Trial 6		Tr	ial 7	Tr	ial 8	Trial 9		Trial 10	
	Na <sub>3</sub> PO <sub>4</sub>	Co(NO <sub>3</sub> ) <sub>2</sub>	Na <sub>3</sub> PO <sub>4</sub>	Co(NO <sub>3</sub> ) <sub>2</sub>	Na <sub>3</sub> PO <sub>4</sub>	Co(NO <sub>3</sub> ) <sub>2</sub>	Na <sub>3</sub> PO <sub>4</sub>	Co(NO <sub>3</sub> ) <sub>2</sub>	Na <sub>3</sub> PO <sub>4</sub>	Co(NO <sub>3</sub> ) <sub>2</sub>
Initial Burette Reading (mL) Final Burette Reading (mL)										
Volume (mL)										

### TABLE 2

	Trial 1	Trial 2	Trial 3	Trial 4	Trial 5
Mass of empty					
filter paper					
(grams)					
Mass of filter					
paper +					
precipitate					
(grams)					
Mass of					
precipitate					
(grams)					

#### TABLE 2 (CONTD.)

	Trial 6	Trial 7	Trial 8	Trial 9	Trial 10
Mass of empty filter paper (grams)					
Mass of filter paper + precipitate (grams)					
Mass of precipitate (grams)					

### Calculations

BASED ON THE DATA OBTAINED IN PART 2 OF THE EXPERIMENT, PERFORM THE FOLLOWING CALCULATIONS FOR EACH TRIAL AND SHOW YOUR WORK IN DETAIL.

a. Calculate the moles of Na<sub>3</sub>PO<sub>4</sub> used in the experiment.

moles of Na<sub>3</sub>PO<sub>4</sub> = Molarity of Na<sub>3</sub>PO<sub>4</sub> × Volume in liters of Na<sub>3</sub>PO<sub>4</sub>

b. Calculate the moles of  $Co(NO_3)_2$  used in the experiment.

moles of  $Co(NO_3)_2$  = Molarity of  $Co(NO_3)_2$  × Volume in liters of  $Co(NO_3)_2$ 

c. Calculate the moles of  $Co(NO_3)_2$  needed (assuming  $Na_3PO_4$  is the limiting reagent)

moles of Co(NO<sub>3</sub>)<sub>2</sub> needed = moles of Na<sub>3</sub>PO<sub>4</sub> (from part a) ×  $\frac{3 \text{ moles Co(NO_3)}_2}{2 \text{ moles Na}_3PO_4}$ 

- Identify the Limiting Reagent by comparing the answers to parts "b" and "c".
  [HINT: If you have less than what is needed, that reagent is the limiting reagen]
- e. Calculate the theoretical yield, in grams, of Co<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub>. [HINT: convert moles of limiting reagent to moles of Co<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub> and then convert moles of Co<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub> to grams of Co<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub>]
- f. What is the actual yield (same as the experimental yield) of  $Co_3(PO_4)_2$ ?
- g. What is the percent yield of this trial?

<u>COLLECT THE FOLLOWING INFORMATION FOR EACH TRIAL FROM YOUR CLASS MATES AND ENTER IN</u> <u>THE FOLLOWING TABLE:</u>

	Mass of Na <sub>3</sub> PO <sub>4</sub> (grams)	Actual Mass of Co <sub>3</sub> (PO <sub>4</sub> ) <sub>2</sub> (grams)	Theoretical Mass of Co <sub>3</sub> (PO <sub>4</sub> ) <sub>2</sub> (grams)
Trial 1			
Trial 2			
Trial 3			
Trial 4			
Trial 5			
Trial 6			
Trial 7			
Trial 8			
Trial 9			
Trial 10			

Create two separate graphs.

1. Plot a graph of the Actual Mass of Co<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub> (y-axis) vs. Mass of Na<sub>3</sub>PO<sub>4</sub> (x-axis).

2. Plot a graph of the Theoretical Mass of  $Co_3(PO_4)_2$  (y-axis) vs. Mass of  $Na_3PO_4$  (x-axis).

3. In the graph that plots Theoretical Mass of  $Co_3(PO_4)_2$  (y-axis) vs. Mass of  $Na_3PO_4$  (x-axis) explain why the graph plateaus. What is the limiting reagent as the slope increases? What is the limiting reagent after the data plateaus?

4. How does the plot of the Actual Mass of  $Co_3(PO_4)_2$  (y-axis) vs. Mass of  $Na_3PO_4$  (x-axis) compare to the plot of the Theoretical Mass of  $Co_3(PO_4)_2$  (y-axis) vs. Mass of  $Na_3PO_4$  (x-axis). Why might these two plots differ?