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Experiment 9
Limiting the Reaction

Learning Objectives:

Upon completion of this experiment, students will:

1. (CLO3). Analyze evidence to decide if generalizations or conclusions based on the obtained data are warranted
2. (CLO4). Interpret and utilize mathematical formulas while solving problems
3. (MLO) Demonstrate an understanding of stoichiometry by calculating theoretical yield, actual yield, percent yield and limiting reagent.
4. Experience using gravity filtration and gravimetric analysis.

Discussion:

The limiting reactant of a reaction is the chemical species which is the first to be completely consumed in the reaction. It is the reactant that limits the amount of products which can be made. As an example, consider the construction of a car from 10 wheels and 2 frames. Only 2 cars can be made with two wheels left over or in excess, making the frames the limiting reactant.

You will prepare a salt mixture of strontium chloride hexahydrate, $SrCl_2{\cdot}6H_2O$, and sodium phosphate dodecahydrate, $Na_3PO_4{\cdot}12H_2O$, and allow them to react in approximately 150 mL of deionized water. Both of these salts are soluble in water but the product, strontium phosphate is not. In the reaction mixture, one of the hydrated salts will limit the amount of strontium phosphate is produced.

Once the precipitation reaction is complete, the precipitate is filtered, dried, and weighed. The precipitate is captured in the filter paper, and the liquid that passes through the filter paper, called the **filtrate,** is collected and tested with very concentrated solutions of the reactants. The extent of the reaction is being tested. For example, if we go back to the production of the cars, we made two cars with 2 wheels left over. If we buy more wheels, we cannot make more cars because we have even more of the wheels in excess; however if we purchase more frames, we could begin to make more cars with the leftover wheels. In this reaction, the reactant that is in excess is determined by testing the filtrate with more of the reactants. The filtrate will clearly show a precipitate if there is excess reactant present with which the testing reactant can react. If strontium chloride is in excess, adding strontium chloride will not show a precipitate, but by adding the reactant, sodium phosphate, which reacts with strontium chloride, the resulting solution will show a precipitate of strontium phosphate. In this reaction, PO_4^{3-} ions react with Sr^{2+} ions in excess producing a precipitate. The same is true if strontium ions are added to a solution that contains phosphate ions. If only one reaction shows a precipitate, the limiting reactant can be determined. If both tubes show cloudiness, it means that the reaction did not go to completion. If neither reaction shows a precipitate, then both reactants were added so that both react completely.

Before the precipitate can be filtered, the precipitate particles must be large enough that they are trapped by the filter paper. Precipitation begins by a process called nucleation in which tiny particles of the precipitate form. These particles are too small to be trapped by the filter paper. The particle's size of the precipitate can be increased through digestion, a gentle heating process. Nucleation is favored in saturated solutions, and particle growth is favored in unsaturated solutions. A saturated solution is one that holds as much solute as possible at the given temperature. An unsaturated solution holds less than the maximum amount of solute. When water is added to the solid salt mixture the resulting solution is relatively saturated, which is not the best situation for creating large precipitate particles that are easy to filter. In order to encourage particle growth the saturation of the solution is decreased by gentle heating.

After the strontium phosphate is filtered, dried, and weighed, the starting mass of the limiting reactant salt can be determined. The calculation of quantities of reactants used and products created can be determined using the process of calculations called ***stoichiometry.*** Stoichiometry relates the mass of each substance to its number of moles and the moles of the reactants to the moles of products using the balanced chemical reaction. This process can be used under any situation where a chemical reaction is known and the quantities measured. Stoichiometry can be shown as

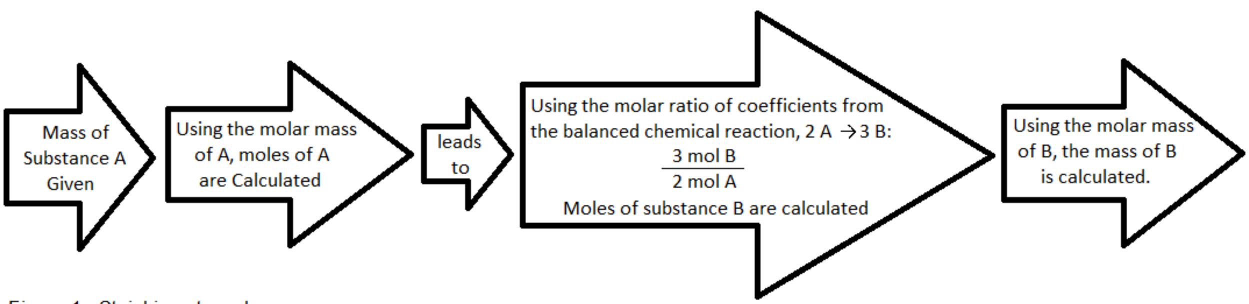

Figure 1: Stoichiometry scheme

$$mass_A \div molar\ mass\ of\ A = moles_A;\ \ mass_A \times \frac{1\ mol}{\mathcal{MM}\ (g)} = \mathrm{mol_A}$$

$$moles_A \times \frac{coefficient\ of\ B\ in\ balanced\ reaction}{coefficient\ of\ A\ in\ balanced\ reaction} = theoretical\ moles_B$$

$$moles_B \times molar\ mass\ of\ B = mass_B;\ \ mol_B \times \frac{\mathcal{MM}(g)}{1\ mole} = theoretical\ yield\ or\ mass_B$$

The first step in any stoichiometry problem is to determine the balanced chemical reaction. In most of the examples you will be given the reaction or the reactants and must determine the overall balanced reaction. In this problem, the two ionic compounds react in a double replacement reaction and the products are easily predicted from the reactants. The mass of a single reactant or in this case the mass of both reactants are then measured. The molar mass of the reactants can be calculated from the atomic masses, thus allowing us to calculate the moles of each reactant. For example if we wish to determine the moles of $CaCl_2$ present in 10.00 g of the salt, then

$$10.00\ g\ CaCl_2 \times \frac{1\ mol\ CaCl_2}{110.984\ g\ CaCl_2} = 0.09010\ mol\ CaCl$$

If $CaCl_2$ is reacted with Na_2SO_4,, the balanced reaction is

$$CaCl_2\ (aq) + Na_2SO_4\ (aq) \rightarrow 2\ NaCl\ (aq) + CaSO_4\ (\text{solid precipitate})$$

Then the mass of the precipitate can be determined as

$$0.09010\ mol\ CaCl_2 \times \frac{1\ mol\ CaSO_4}{1\ mol\ CaCl_2} = 0.09010\ mol\ CaSO_4$$

And the **theoretical mass** of the precipitate calculated using the molar mass of the calcium sulfate as

$$0.09010\ mol\ CaSO_4 \times \frac{136.141\ g\ CaSO_4}{1\ mol\ CaSO_4} = 12.27\ g\ CaSO_4$$

If a sample of mixed $Na_2SO_4$and $CaCl_2$ with a mass of 1.017 g was dissolved, digested, and filtered, can we determine the percent of the original sample that was the limiting reactant? First we have to determine which substance was limiting by the reagent test. Precipitation tests on the supernatant determined that Na_2SO_4 was the limiting reactant. The precipitate was collected on a 5.000 g piece of filter paper. Once dried, the filter paper and precipitate has a combined mass of 5.198 g.

The mass of calcium sulfate is 0.198 g, or 1.45×10^{-3} mol $CaSO_4$. The precipitation test on the supernatant lets us know that the sodium sulfate is limiting, which means that all of the sulfate is present in the precipitated calcium sulfate. This means that the moles of Na_2SO_4 can be determined from the moles of precipitate. Note that the moles of calcium *cannot* be determined from the moles of calcium sulfate, since the calcium chloride was in excess, there is excess calcium ions in the supernatant. Starting from the moles of calcium sulfate precipitate, we calculate the moles of sodium sulfate in the original salt mixture:

$$0.198\ g\ CaSO_4 \times \frac{1\ mol\ CaSO_4}{136.141\ g\ CaSO_4} \times \frac{1\ mol\ Na_2SO_4}{1\ mol\ CaSO_4} = 0.00145\ mol\ Na_2SO_4$$

$$0.00145\ mol\ Na_2SO_4 \times \frac{142.042\ g\ Na_2SO_4}{1\ mol\ Na_2SO_4} = 0.206\ g\ Na_2SO_4$$

From this, the mass percent of limiting reactant can be calculated:

$$\frac{0.206\ g\ Na_2SO_4}{1.017\ g\ sample}\times 100 = 20.3\%\ Na_2SO_4$$

The mass of the in excess reactant can be determined by difference. If 1.017 g of the mixed salts contained 0.206 g was the limiting reactant, then 0.811 g of $CaCl_2$ which is 79.7% of the mixture.

Procedure:

Label two beakers Beaker I and Beaker II.

In the beaker labeled Beaker I, separately weigh 1.0 g of $Na_3PO_4\cdot 12H_2O$ and 1.5 g of $SrCl_2\cdot 6H_2O$, record the exact mass of each salt added to the beaker.

In the beaker labeled Beaker II, weigh approximately 2 g of the solid $Na_3PO_4\cdot 12H_2O$ - $SrCl_2\cdot 6H_2O$ **mixture provided**, record the exact mass of the mixture added to the beaker.

The procedure for each beaker is the same at this point.
Use the following procedure for both Beaker I and Beaker II.

Add approximately 150 mL of deionized water to the beaker while stirring, continue stirring for approximately 3 minutes. Leave the stir rod in the beaker, cover with a watch glass, and allow the undissolved salts or precipitate to settle to the bottom of the beaker.

Digest the precipitate for approximately 15 minutes using a hot plate on low heat or place the beaker on the ring stand over a Bunsen burner with a cool blue flame. Do not let the mixture come to a boil. Remove the heat from the beaker if the mixture begins to boil.

After 15 minutes of digestion, remove the beaker containing the sample from the heat and let it stand until the mixture and beaker is cool enough to be safely handled. Heat about 100 mL of deionized water to between 70 - 80°C. This water will be used for washing the precipitate out of the beakers and off the stir rods. This water will also be used to rinse the precipitate in the funnel.

While the sample cools and the wash water is heating, set up the filtration apparatus. The filtration can be done by gravity filtration or by vacuum filtration. Obtain one sheet of fine porosity filter paper for each beaker. Using pencil, label the filter paper with your initials and sample number. Record the mass of each dry filter paper. Filter the sample and set the filtrate aside for testing. Transfer all of the precipitate into the funnel, rinse the precipitate with the warmed wash water. **DO NOT throw away the filtrate.**

Place the filter paper on a clean watch glass to dry. The sample can be dried in a drying oven to a constant mass of ± 0.01 grams or placed in a safe place until the next laboratory period. Allow the filtrate to settle and carefully decant 5 mL of the filtrate into two test tubes, labeled $SrCl_2$ and Na_3PO_4. If the filtrate is still slightly cloudy, centrifuge the samples or allow the precipitate to settle to the bottom of the test tube. Add 5 drops of 1M $SrCl_2$ test reagent to tube labeled $SrCl_2$ to test for the presence of phosphate ion in excess and 5 drops of 1 M Na_3PO_4 reagent to test tube labeled Na_3PO_4 to test for excess strontium ions. You should see an obvious precipitation reaction in only one of the tubes. Dispose of the filtrate in the liquid waste container and clean your glassware. Complete the procedure with the second beaker. Once your samples are dry, record the mass of each sample and determine the mass of the precipitate collected. Using the final mass, complete the calculations for the laboratory assignment.

Two lab groups can work together, one completing the procedure for Beaker 1 and one completing the procedure for Beaker II. The data can then be collected and distributed to the whole class or the two groups can share their data. All calculations must be completed for both beakers.

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Pre Laboratory Problems

1. Write a purpose statement and a summary of the procedure to include special techniques, equipment, chemicals, chemical reactions to be performed, an understanding of any calculations that are important to understanding the procedure.

2. Calculate the molar mass of the following compounds.
   a. strontium chloride hexahydrate, $SrCl_2{\cdot}6H_2O$,

   b. sodium phosphate dodecahydrate, $Na_3PO_4{\cdot}12H_2O$ and

   c. Strontium phosphate $Sr_3(PO_4)_2$

3. When a hydrated salt dissolves in water, what happens to the water in the salt?

4. What is the overall balanced reaction when an aqueous solution of $SrCl_2$ reacts with an aqueous solution of Na_3PO_4?

5. 2.000 g of $Na_3PO_4{\cdot}12H_2O$ is reacted with 2.000 g of $SrCl_2{\cdot}6H_2O$, calculate the theoretical yield of $Sr_3(PO_4)_2$ precipitate. Complete the following steps to solve this problem.

   a. Calculate the theoretical yield (mass) of $Sr_3(PO_4)_2$ produced from 2.000 g of $Na_3PO_4{\cdot}12H_2O$ if we assume that the strontium chloride salt is in excess.

b. Calculate the theoretical yield of $Sr_3(PO_4)_2$ produced from 2.000 g of $SrCl_2{\cdot}6H_2O$ assuming that the sodium phosphate is in excess

c. Which reactant produces the smallest theoretical yield?

d. Which compound is the limiting reactant?

6. In the procedure, the filtrate is tested with the two test reagents, what does it mean if the test tube treated with 1 M $SrCl_2$ reagent shows a precipitate?

7. A 1.500 g sample of a mixture of solid $Na_3PO_4{\cdot}12H_2O$ mixed with NaCl, was dissolved in water, reacted with aqueous 1 M $SrCl_2$ and 0.520 g $Sr_3(PO_4)_2$ precipitate was collected and dried to a constant mass. If the reagent tests demonstrated that $Na_3PO_4{\cdot}12H_2O$ was the limiting reactant in the precipitation reaction, calculate the mass percent of $Na_3PO_4{\cdot}12H_2O$ in the original sample.

   a. How many moles of $Sr_3(PO_4)_2$ were produced?

   b. How many moles of Na_3PO_4 were present in the aqueous solution?

   c. If the moles of Na_3PO_4 are equivalent to the moles of $Na_3PO_4{\cdot}12H_2O$, what is the theoretical mass of $Na_3PO_4{\cdot}12H_2O$ in the solid mixture?

   d. What is the mass percent of sodium phosphate dodecahydrate in the solid mixture?

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Laboratory Report

Beaker Sample I

1.	a.	Mass of $SrCl_2 \cdot 6H_2O$ and container	**0.0000 g**
	b.	Mass of container	**0.0000 g**
	c.	Mass of $SrCl_2 \cdot 6H_2O$ added to Beaker I	
	d.	Moles of $SrCl_2 \cdot 6H_2O$ added to Beaker I	
	e.	Theoretical moles of $Sr_3(PO_4)_2$ determined from moles of $SrCl_2 \cdot 6H_2O$	
2.	a.	Mass of $Na_3PO_4 \cdot 12H_2O$ with container	**0.0000 g**
	b.	Mass of container	**0.0000 g**
	c.	Mass of $Na_3PO_4 \cdot 12H_2O$ added to Beaker I	
	d.	Moles of $Na_3PO_4 \cdot 12H_2O$ added to beaker I	
	e	Theoretical moles of $Sr_3(PO_4)_2$ determined from moles of $Na_3PO_4 \cdot 12H_2O$	
3.		Theoretical moles of $Sr_3(PO_4)_2$ in Beaker I determined from the limiting reagent (1e or 2e)	
4.		Theoretical yield of $Sr_3(PO_4)_2$ in grams	
5.	a.	Observation of 1 M $SrCl_2$ test reagent, added to filtrate from Beaker I	
	b.	Observation of 1 M Na_3PO_4 test reagent added to filtrate	
	c.	Formula of limiting reactant	
	d.	Formula of excess reactant	

6. a. Mass of precipitate and paper First weighing ____________________

Second weighing ____________________

Third weighing ____________________

b. Mass of filter paper ____________________

c. Actual yield of $Sr_3(PO_4)_2$ ____________________

d. Percent Yield of $Sr_3(PO_4)_2$ ____________________

7. Clearly show all calculations for the above procedure (be sure to include units).

8. The limiting reactant can be determined from the calculation of the theoretical yield of the product. Why is it necessary to test the filtrate with a concentrated solution of the reagents?

Beaker Sample II

1. Identity of Unknown mixture ____________________

2. a. Mass of mixture and container ____________________

b. Mass of container ____________________

c. Mass of mixture added to Beaker II ____________________

3. a. Mass of precipitate and paper First weighing ____________________

Second weighing ____________________

Third weighing ____________________

b. Mass of filter paper ____________________

c. Actual yield of $Sr_3(PO_4)_2$ ____________________

4. From the actual yield, calculate the moles of $Sr_3(PO_4)_2$ ____________________

5. a. Observation of $SrCl_2$ test reagent, added to filtrate from Beaker II

b. Observation of Na_3PO_4 test reagent added to filtrate

c. Formula of limiting reactant

d. Formula of excess reactant

6. a. From the actual moles of product determined above, calculate the moles of limiting reactant in sample

b. Theoretical mass of limiting reactant

c. Mass percent of limiting reactant in sample

7. a. Mass of in excess reactant in sample

b. Mass Percent of excess reactant in sample

8. Explain how you determined the mass percent of each reactant in the sample (calculations can be used to show how you determined these values.)

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Post Laboratory Problems and Conclusions

1. In the pre laboratory problems, you determined the mass of precipitate formed when equal amounts of the reactants were used. Explain the reason why the reactants do not produce the same amount of product, why does one reactant limit the reaction and the other reactant is in excess.

2. How do the coefficients of the reaction relate to the amounts of the reactants used?

3. Why is it important to understand stoichiometry, limiting and excess reactants, theoretical yield, actual yield and % yield?

4. Write a conclusion paragraph on what you learned and why these techniques are important to science.