

## Experiment 1: Determining the Densities of Solids

### Theory

Density measures the ratio of the mass of a substance to its volume. It is a physical property of any substance and it is unique to that substance. Density can be used to identify and also determine how pure a substance is. Another way density is useful to man is that it helps to know whether a substance will float or sink when immersed in a liquid. For example, if a substance is less dense than a liquid, then that substance will float and vice versa. That is why a boat or ship will float on water because it is less dense than water.

By Archimedes principle, a substance, when immersed in a liquid can only displace an amount of liquid that is equal to its own mass. Therefore, we can easily determine the density of any substance, by finding the ratio of its mass to its volume

$$\text{Density} = \text{Mass of a substance in grams} / \text{Volume of substance in cm}^3 \text{ (mL)}$$

### Experiment: To determine the density of a Solid

**Apparatus:** Analytical Balance, 3 solid materials (A, B C), three 10 mL measuring cylinders, water.

**Procedure:** Press the **ON** button to turn the balance on. Zero the balance by pressing the **Zero** button. Choose the units your desire by pressing the **Unit** button. Label the three solids as **A, B, C** and the measuring cylinder as **D, E, F** respectively. Place cylinder **D** on the balance. Allow the balance to stabilize and record the weight in **Table 1**. Add 20.0 mL of water to the cylinder and then record the new weight. Lastly, gently lower solid **A** into the cylinder and then record the final weight and the new volume of water. Remove cylinder **D** and repeat the same procedure for solids **B** and **C** using cylinders **E** and **F** respectively. Dry out the solids and the measuring cylinders and return them to their appropriate places.

Complete the Table and report your answer to 2 decimal places.

**Experiment 1****RESULT****Name****Department****Date****Table 1**

	<b>Test</b>	<b>A</b>	<b>B</b>	<b>C</b>
1	Mass of Cylinder			
2	Mass of Cylinder + 5 mL of Water			
3	Mass of Cylinder + 5 mL of Water + Solid			
4	New volume of water (lower meniscus of water)			
5	Mass of Solid (Line 3 - Line 2)			
6	Volume of Solid (Line 4 - 5 mL)			
7	Density of Solid( Line 5 divided by Line 6)			

1. Can you identify the solids used in this experiment?
2. Calculate the density (g/mL) of mercury if 9.75L weighs 132.5 kg

## Experiment 2: Determining the density of a Liquid

### Theory

Production of energy is very essential in building a sustainable economy. Crude oil is the most common source of energy used in all parts of the world. Crude oil is very important and it plays a very crucial part both in the economy and the politics of our present time. Nigeria is the seventh largest producer of crude oil in the world. Crude oil is extracted from the ground and transported in tanks by ships and trucks to the refinery. Along the line, spillages occur and the environment becomes highly polluted. For example, the Niger-Delta region of Nigeria produces crude oil and it's highly polluted due to oil spillage. Crude oil contains mostly hydrocarbons (compounds containing carbon and hydrogen) whose densities are mostly less than 1 (density of water is 1.0 g/mL) and hence it will float on water whenever there is spillage during transportation by ship or in the case of off shore drilling (which is the case in the Niger-Delta of Nigeria). Such spillages into the water ways and the environment are a great health hazard especially to the marine, inland ecosystem and the human health. It is good to determine the densities of the different hydrocarbon fraction present in the crude oil so as to know which fraction will float and which will sink to the bottom of the ocean and adapt the best method of cleaning.

Density =

$$\frac{\text{Mass of a Liquid in grams}}{\text{Volume of Liquid in cm}^3 \text{ (mL)}}$$

**Apparatus:** Analytical Balance, 3 liquids (G, H, I), three 10 mL measuring cylinders.

### Procedure

#### Part1: Calibration of a Container.

Press the **ON** button to turn the balance on. Zero the balance by pressing the **Zero** button. Choose the units your desire by pressing the **Unit** button. Weigh a dry and clean 10mL measuring cylinder to the nearest 0.01g. Fill the cylinder with water and adjust the lower meniscus to exactly 10.0 mL with a pipet. Reweigh the measuring cylinder using the same balance and record your data in **Table 2a**. Calculate the volume to the cylinder to the nearest 0.1 mL by using the density of water (1.0 g/mL at room temperature). Empty the cylinder and repeat the procedure two more times. Find the average of the volume of water and approximate to two significant figures. Dry the cylinder by using 2 mL of acetone. Empty the acetones into the bottle provided and allow the cylinder to dry for a few minutes.

#### Part 2: Density of a Liquid

Label the three liquids as **GHI** and the measuring cylinder as **J, K, L** respectively. Place cylinder **J** on the balance. Allow the balance to stabilize and record the weight in **Table 2b**. Add 5.0 mL of Liquid **G** to cylinder **J** and then record the new weight. Remove cylinder **J** and repeat the same procedure for liquids **H** and **I** using cylinders **K** and **L** respectively. Return the liquids to their respective containers. Clean and dry the measuring cylinders using water and acetone. Return the measuring cylinders to their appropriate places. Complete Tables 2a and 2b and report your answer to 2 decimal places.

## Experiment 2: Determining the density of a Liquid

### Theory

Production of energy is very essential in building a sustainable economy. Crude oil is the most common source of energy used in all parts of the world. Crude oil is very important and it plays a very crucial part both in the economy and the politics of our present time. Nigeria is the seventh largest producer of crude oil in the world. Crude oil is extracted from the ground and transported in tanks by ships and trucks to the refinery. Along the line, spillages occur and the environment becomes highly polluted. For example, the Niger-Delta region of Nigeria produces crude oil and it's highly polluted due to oil spillage. Crude oil contains mostly hydrocarbons (compounds containing carbon and hydrogen) whose densities are mostly less than 1 (density of water is 1.0 g/mL) and hence it will float on water whenever there is spillage during transportation by ship or in the case of off shore drilling (which is the case in the Niger-Delta of Nigeria). Such spillages into the water ways and the environment are a great health hazard especially to the marine, inland ecosystem and the human health. It is good to determine the densities of the different hydrocarbon fraction present in the crude oil so as to know which fraction will float and which will sink to the bottom of the ocean and adapt the best method of cleaning.

Density =

$$\frac{\text{Mass of a Liquid in grams}}{\text{Volume of Liquid in cm}^3 \text{ (mL)}}$$

**Apparatus:** Analytical Balance, 3 liquids (G, H, I), three 10 mL measuring cylinders.

### Procedure

#### Part1: Calibration of a Container.

Press the **ON** button to turn the balance on. Zero the balance by pressing the **Zero** button. Choose the units your desire by pressing the **Unit** button. Weigh a dry and clean 10mL measuring cylinder to the nearest 0.01g. Fill the cylinder with water and adjust the lower meniscus to exactly 10.0 mL with a pipet. Reweigh the measuring cylinder using the same balance and record your data in **Table 2a**. Calculate the volume to the cylinder to the nearest 0.1 mL by using the density of water (1.0 g/mL at room temperature). Empty the cylinder and repeat the procedure two more times. Find the average of the volume of water and approximate to two significant figures. Dry the cylinder by using 2 mL of acetone. Empty the acetones into the bottle provided and allow the cylinder to dry for a few minutes.

#### Part 2: Density of a Liquid

Label the three liquids as **GHI** and the measuring cylinder as **J, K, L** respectively. Place cylinder **J** on the balance. Allow the balance to stabilize and record the weight in **Table 2b**. Add 5.0 mL of Liquid **G** to cylinder **J** and then record the new weight. Remove cylinder **J** and repeat the same procedure for liquids **H** and **I** using cylinders **K** and **L** respectively. Return the liquids to their respective containers. Clean and dry the measuring cylinders using water and acetone. Return the measuring cylinders to their appropriate places. Complete Tables 2a and 2b and report your answer to 2 decimal places.

**Experiment 2****RESULT**

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**Table 2a**

	Test	A	B	C	Average
1	Mass of Cylinder				Nil
2	Mass of Cylinder + 10 mL of water				Nil
3	Mass of Liquid (Line 2 - Line 1)				Nil
4	Volume of Water				
6	Density of Water	1.0	1.0	1.0	1.0

**Table 2b**

	Test	G	H	I
1	Mass of Cylinder			
2	Mass of Cylinder + 10 mL of liquid			
3	Mass of Liquid (Line 2 - Line 1)			
4	Volume of Liquid			
5	Density of Solid( Line 3 divided by Line 4)			

1. Can you identify the liquids used in this experiment?
2. Calculate the density (mg/mL) of mercury if 9.75L weighs 132.5 kg
3. What is the size of 1 quart (946 mL) of corn oil if its density is 0.992 g/mL
4. An empty 25 mL cylinder weighs 54.25 g, and weighs 79.55 g when filled with water. When filled to exactly the same volume with kerosene, the weight is 75.00 g. calculate the density of the kerosene. (Density of water = 1.0 g/mL)

### Experiment 3: Separation of a Mixture

#### Theory

A mixture is made from two or more pure substances that are physically mixed together. Each of the substance in the mixture will still retains its chemical identity. A good example is when water is mixed with salt or pebbles are mixed with rice. Each substance will remain in the same pure form as it were before the mixing process took place. There are two types of mixtures: (a) homogeneous mixture which has a uniform composition and properties and is also referred to as a **solution**. Examples include a mixture of water and salt or a brewed coffee or tea; (b) Heterogeneous mixture have different composition and properties at different regions of the mixture e.g. mixture of rice and pebble, brass (made of zinc and copper).

Most substances in nature are found as mixtures and will need different chemical or physical process to separate them into their pure forms. This experiment uses a combination of chemical and physical properties to separate a heterogeneous mixture of sodium chloride, calcium carbonate and sand. Sodium Chloride is soluble in water and can be washed out of the mixture; Calcium carbonate can dissolve in acid to give carbon dioxide (a gas) and calcium chloride which can be washed out of the mixture with water. At the end of these two processes, only sand will be left in the container in its pure form.

**Apparatus:** Evaporating dish, Weighing balance, stirrer, measuring cylinder, tripod stand, wire gauze burner (oven), crucible tongs, desiccators, 1 M HCl, water, mixture of unknown composition.

**Procedure:** Weigh a very clean and dry evaporating dish and record its weight in **Table 3**. Then, weigh 5g ( $\pm 0.001$ g) of a mixture of unknown composition with the evaporating dish. Add 25 mL of water to the mixture and stir and then allow the solids to settle out. Carefully pour out the water not losing any solid. **Repeat the process three more times.** Set up a tripod, wire gauze and the burner and heat the dish for 5 minutes to dry the mixture. Use the tongs to place the heated dish in a desiccator and allow it to cool for 15-20 minutes. Reweigh the dish. Heat the dish again for two minutes and allow it to cool and then reweigh. Repeat the two minutes heating and cooling process until the succeeding weights differ by 2 % or less. Record your final weight in Table 3

Slowly add 25 mL of 1M HCl to the dish and stir gently for 2 minutes. Gently pour of the liquid into a provided container and repeat the process two more times with 10 mL of the acid each time. Wash the remaining solids with 10 mL of water. Repeat the water wash two more times and then heat the remaining solid to dryness checking for succeeding weight to be within 2% of each other.

**Special Waste:** Place any leftover unknown, isolated sand, and the acid in the specially marked container.

**Cautions:** Handle the hot evaporating dishes with tongs. Make sure all gas jets are turned off before leaving the laboratory.

### Experiment 3

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Table 3

Unknown	Weights (g)
Mass of evaporating dish	
Mass of evaporating dish and unknown	
Mass of Unknown	
<b>Sodium Chloride NaCl</b>	
Mass of evaporating dish and dried mixture (Final Weighing after washes)	
Mass loss due to NaCl	
% of NaCl in unknown	
<b>Calcium Carbonate CaCO<sub>3</sub></b>	
Mass of dish plus Sand (Final weighing after washes)	
Mass loss due to CaCO <sub>3</sub>	
% of CaCO <sub>3</sub> in unknown	
<b>Sand</b>	
Mass of SiO <sub>2</sub>	
% of SiO <sub>2</sub> in unknown	
<b>Total of Percentages</b>	

1. If the % does not tally to 100%, give reasons for the difference.
2. In this experiment, what property of NaCl is used to separate it from the original mixture? Is this a chemical or physical property?
3. If the original mixture weighed 6.40 g and lost 1.85 g during the water wash, what is the % of NaCl by mass?
4. If the mixture weighed 2.05 g after the acid wash, what are the %'s of CaCO<sub>3</sub> and SiO<sub>2</sub> by Mass?
5. What would be the effect on the percentage of each of the three components if, during the first washes, the fine particles of calcium carbonate are not allowed to settle out before pouring off the water?

## Experiment 4: Chemical Periodicity

### Theory

The periodic table is formed by arranging elements in a certain way that shows a specific periodic variation. When the elements are arranged in increasing atomic number the result is the formation of period (horizontal) and group (vertical) with similar variation and trends in their physical and chemical properties. An example of the periodicity obtained by this arrangement is seen in group IIa alkaline earth metals where every element in that group possesses  $ns^2$  electrons in its outermost shell and also form compounds with the same general formula (forms chlorides with a general formula  $RCl_2$ ). Other examples of similar trends include observation of increasing atomic sizes down a group while ionization energy or electronegativity decreases down a group.

This experiment will demonstrate variation in solubilities of the alkaline earth metals (group IIA) by observing if precipitates are formed or not when these compounds are formed during a reaction. All other reactants and products in these reactions are known to be soluble.

**Apparatus:** Four test tubes, Droppers

**Reagents:** 0.1 M  $K_2CrO_4$ , 0.1M  $Mg(NO_3)_2$ , 0.1M  $Ca(NO_3)_2$ , 0.1M  $Sr(NO_3)_2$ , 0.1M  $Ba(NO_3)_2$ , 0.1M  $H_2SO_4$ , 0.25M  $NH_4C_2O_4$ , 1.0M  $Na_2CO_3$

### Procedure

Label four test tubes as **ABCD** and add 10 drops of 0.1 M  $K_2CrO_4$  to each of them. Add 10 drops of 0.1M  $Mg(NO_3)_2$ , 0.1M  $Ca(NO_3)_2$ , 0.1M  $Sr(NO_3)_2$ , 0.1M  $Ba(NO_3)_2$  to test tubes **ABCD** respectively. Observe which of the reactions produces a precipitate and those that do not produce a precipitate. Clean the test tube and rinse thoroughly with water. Repeat the experiments using 0.1M  $H_2SO_4$ , 0.25M  $NH_4C_2O_4$ , 1.0M  $Na_2CO_3$  each time adding the four alkaline earth metals. Record all your observations in the table shown below

**Experiment 4****RESULT****Name****Department****Date****Table 4**

	<b>0.1 M K<sub>2</sub> CrO<sub>4</sub></b>	<b>0.1M H<sub>2</sub>SO<sub>4</sub></b>	<b>0.25M NH<sub>4</sub>C<sub>2</sub>O<sub>4</sub></b>	<b>1.0M Na<sub>2</sub>CO<sub>3</sub></b>
<b>0.1M Mg(NO<sub>3</sub>)<sub>2</sub></b>				
<b>0.1M Ca(NO<sub>3</sub>)<sub>2</sub></b>				
<b>0.1M Sr(NO<sub>3</sub>)<sub>2</sub></b>				
<b>0.1M Ba(NO<sub>3</sub>)<sub>2</sub></b>				

**Question**

1. Is there any trend in solubility down the group? If so what is the trend?
2. What do the elements in a single group have in common regardless of their period?

## Experiment 5: ACID BASE TITRATIONS

### Theory

The reaction of an acidic solution with a basic solution can be represented by the net ionic equation



The reaction changes gradually from an acidic or basic solution or vice versa to a neutral solution and therefore, the process is called **Neutralization** process. This reaction proceeds to completion and is quantitative; hence, it can be used to calculate the concentration of an acid or a base. The procedure of gradually adding an acid to a base or vice versa to form a neutral solution is also called **Titration**. So, if the concentration of the acid or base is known, then the concentration of the other one can be calculated by determining the **equivalent point** or **end point** (concentration of acid = concentration of base) in the process of neutralization (titration). To determine the end point an indicator is added before titration starts. An indicator usually is a weak organic acid which changes its color at a specific pH.

At the end point, the amount of acid and the base are equal, therefore,

$$\text{The number of Moles (mol) acid} = \text{the number of Moles (mol) of base (1)}$$

When we use **molarity (M)** to express the concentration of the acid and base, **then**

$$\text{M} = \text{mol/L or M} = \text{mmol/mL and, (2)}$$

$$\# \text{ of mol} = (\text{M})(\text{L}) \text{ or } \# \text{ of mmol} = (\text{M})(\text{mL}) \quad (3)$$

We can then say that at equivalent or end point, equation becomes

$$(\text{M}_{\text{acid}})(\text{L}_{\text{acid}}) = (\text{M}_{\text{base}})(\text{L}_{\text{base}}) \text{ or}$$

$$(\text{M}_{\text{acid}})(\text{mL}_{\text{acid}}) = (\text{M}_{\text{base}})(\text{mL}_{\text{base}})$$

Finally,

$$\text{M}_{\text{base}} = (\text{M}_{\text{acid}})(\text{L}_{\text{acid}}) / \text{mL}_{\text{base}}$$

Acids have replaceable hydrogen's which takes part in its chemical reaction. The number of such replaceable hydrogen's determines the **equivalent weight** of the acid. The **equivalent weight** of an acid is then defined as the weight of the acid that will react with one mole of NaOH or other monobasic substances. If an acid has one replaceable hydrogen atom, then, its equivalent weight will be the same as its molecular weight. If the acid contains two replaceable hydrogen atoms, then its equivalent weight will be half its molecular weight, and so on and so forth.

$$\text{Eq. Wt} = \text{amount of acid / moles of base}$$

## Experiment 5 Con'td

**Apparatus:** 2 Burette, pipet, 2x 250 mL conical flask, 600 mL beaker, brush, stirring rod.  
Retort stand

**Reagent:** Sodium hydroxide (NaOH), Hydrochloric acid (HCl), methyl orange indicator, distilled water

### Standardization of Sodium Hydroxide (NaOH)

**Procedure:** In this experiment, we are going to standardize sodium hydroxide. Wash the beakers, burettes and the flask with soap and water and rinse thoroughly with distilled water. Allow the containers to dry. Use the 600 mL beaker to obtain 10 mL of 3M NaOH from the instructor and add 290 mL of distilled water. Stir the solution with the stirring rod for about 1 minute. Obtain 80 mL of 0.05 M of HCl from the instructor and use 5 mL to rinse the burettes. Attach the burette to the retort stand and fill it to the zero mark. Drain a little bit of the acid in the burette to remove any air bubble in the system. Stir the NaOH solution again and fill the other burette to the zero mark. Drain the NaOH burette to remove air bubbles. Record the initial volumes of the acid and the base using the lower meniscus of the liquids.

Pour 25 mL of distilled water in a 250 ml conical flask and add 3-5 drops of the methyl orange indicator. Drain 15 mL of the HCl from the burette into the flask and stir very well. Carefully and slowly drain NaOH from the burette into the acid solution while stirring at the same time. The acid solution will turn pink and disappear as you stir it. As you get nearer the end-point, the pink color will stay longer. Continue to add NaOH drop by drop at this point until the color does not fade away again. This is the end-point. If you added too much base, add 2-3 drops of acid and then neutralize with NaOH from the burette to reach a new end-point. Record the volume of the base from your burette using the lower meniscus.

Repeat the titration 2 more times.

Clean your entire container and return them to the instructor.

**Do not discard the NaOH solution. It will be used in the next experiment**

**Waste:** Flush all waste through the drain

**Experiment 5****RESULT**

Name

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Date

**Table 5**

	Result 1	Result 2	Result 3
Initial Buret reading, HCl			
Final buret reading, HCl			
Volume used, HCl			
Molarity of HCl			
Initial buret reading, NaOH			
Final buret reading, NaOH			
Volume used, NaOH			
Molarity of NaOH			
Average Molarity of NaOH			

**Question**

1. What volume of 2.0 M NaOH would be needed to make a 200mL of 0.10 M NaOH?
2. What volume of 0.10M NaOH is needed to neutralize 20 mL of 0.10 M HCl?
3. What is the concentration of formic acid solution if a 20 mL sample of the acid needs 15 mL of 0.08 M NaOH?

## Experiment 6: Standardization of An Acid Solution

### Theory

This is a continuation of experiment 5 on acids and bases.

**Apparatus:** 2 Burette, pipet, 2x 250 mL conical flask, 600 mL beaker, brush, stirring rod.  
Retort stand

**Reagent:** Sodium hydroxide (NaOH), Hydrochloric acid (HCl), methyl orange indicator, distilled water.

### Standardization of Acetic Acid ( $\text{CH}_3\text{COOH}$ )

**Procedure:** In this experiment, we are going to standardize an acid solution. Wash the beakers, burettes and the flask with soap and water and rinse thoroughly with distilled water. Allow the containers to dry. Set up the burets and rinse one with 5 mL of the NaOH solution prepared from experiment 5, then, fill it up to the zero mark with NaOH. Obtain 80 mL of acetic from the instructor and rinse the other buret with 5 mL of acetic acid and then fill it up with acetic acid to the zero mark.

Pour 25 mL of distilled water in a 250 ml conical flask and add 3-5 drops of the methyl orange indicator. Drain 15 mL of the acetic acid from the burette into the flask and stir very well. Carefully and slowly drain NaOH from the burette into the acid solution while stirring at the same time. The acid solution will turn pink and disappear as you stir it. As you get nearer the end-point, the pink color will stay longer. Continue to add NaOH drop by drop at this point until the color does not fade away again. This is the end-point. If you added too much base, add 2-3 drops of acid and then neutralize with NaOH from the burette to reach a new end-point. Record the volume of the base from your burette using the lower meniscus.

Repeat the titration 2 more times.

Clean your entire container and return them to the instructor

**Waste:** Flush all waste through the drain

**Do not retain any solution at this time.**

**Experiment 6****RESULT**

Name

Department

Date

**Table 6**

	Result 1	Result 2	Result 3
Initial Buret reading, NaOH			
Final buret reading, NaOH			
Volume used, NaOH			
Molarity of NaOH (from Experiment 5)			
Initial buret reading, Acetic Acid			
Final buret reading, Acetic Acid			
Volume used, Acetic Acid			
Molarity of Acetic Acid			
Average Molarity of Acetic Acid			

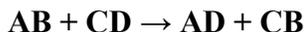
**Question**

1. What volume of 1.6 M NaOH would be needed to make a 500mL of 0.10 M NaOH?
2. What volume of 0.18M NaOH is needed to neutralize 25mL of 0.15 M acetic acid?
3. if 27.66 ml of 0.12M of NaOH is needed to neutralize a sample of unknown acid that weighs 0.35 g, what is the equivalent weight of the acid?

## Experiment 7: Redox and Other Chemical Reactions

### Theory

Double displacement reaction simply involves exchange of ions which can be represented in the following equation.



Acids, bases and salts commonly undergo displacement reactions because they dissociate in aqueous medium. The dissociation can be represented thus;

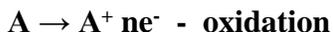


Some of the ions may appear in the same form on both sides of the equation and therefore, omitted from the net ionic equations as shown below



There are evidences that show that displacement reaction has occurred. For example, precipitates may be formed, gas may be evolved (bubbles), or heat may be absorbed or generated. Absence of any of these evidences may indicate that the possible products are soluble in aqueous solution; hence no net reaction has occurred.

**Redox Reaction.** Redox is an abbreviation for reduction and oxidation reaction. Both occurs simultaneously and abound in nature. In redox reactions, electrons are transferred unlike during displacement reactions where ions are transferred. In essence, what is happening is that one species of reagent loses electron while the other gain the same electron at the same time. So, equal moles of electrons is lost and gained at the same time. Redox reaction can then be represented by two half equations thus

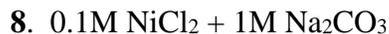
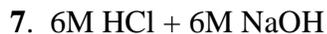


**Apparatus:** Test tubes, droppers

**Procedure:** Mix five drops of each of the following reagent(s) in a test tube. Mix well and note your observation in the Table 7.1

1. 0.25M (NH<sub>4</sub>)<sub>2</sub> C<sub>2</sub>O<sub>4</sub> + 0.1M CoCl<sub>2</sub>
2. 0.1M Ca (NO<sub>3</sub>)<sub>2</sub> + 0.1M NiCl<sub>2</sub>
3. 0.1M Ca (NO<sub>3</sub>)<sub>2</sub> + 1M Na<sub>2</sub>CO<sub>3</sub>
4. 0.1M CuSO<sub>4</sub> + 0.1M Na<sub>3</sub>PO<sub>4</sub>
5. 0.1M Fe(NO<sub>3</sub>)<sub>3</sub> + 6M NaOH
6. 6M HCl + 1M Na<sub>2</sub>CO<sub>3</sub>

## Experiment 7 Con'td



Use Table 7.3 in Appendix A as a guide in determining what type of reaction is occurring in these reactions and the net ionic equation for these reactions (if any)

### Redox Reactions

Mix the following reagents in the order shown in the equations below

1.  $\text{Cu} + 6\text{M HNO}_3$
2.  $\text{Mg} + 6\text{M HCl}$
3.  $0.1\text{ M KMnO}_4 + 6\text{M HCl}$
4.  $0.1\text{ M KMnO}_4 + 6\text{M H}_2\text{SO}_4 + \text{Fe SO}_4$

Record your observations and the net ionic equation in Table 7.2

### Questions

1. After adding five drop so 6M Nitric acid are added to an equal volume of  $\text{Ba(OH)}_2$ , the resulting solution becomes warmer. Write a balanced net ionic equation to describe the reaction.
2. A strip of aluminum metal was dropped in 6M HCl and bubble so Hydrogen gas was observed. The resulting solution contains Aluminum ions ( $\text{Al}^{3+}$ ). Write the net ionic equation for this reaction

**Experiment 7****RESULT**

Name

Department

Date

**Table 7.1**

<b>Reagents</b>	<b>Evidence of Reaction</b>	<b>Net Ionic Equation</b>
$(\text{NH}_4)_2 \text{C}_2\text{O}_4 + \text{CoCl}_2$		
$\text{Ca} (\text{NO}_3)_2 + \text{NiCl}_2$		
$\text{Ca} (\text{NO}_3)_2 + \text{Na}_2\text{CO}_3$		
$\text{CuSO}_4 + \text{Na}_3\text{PO}_4$		
$\text{Fe}(\text{NO}_3)_3 + \text{NaOH}$		
$\text{HCl} + \text{Na}_2\text{CO}_3$		
$\text{HCl} + \text{Na OH}$		
$\text{NiCl}_2 + \text{Na}_2\text{CO}_3$		

## Experiment 7 Con'td

Table 7.2

Reagents	Evidence of Reaction	Net Ionic Equation
$\text{Cu} + \text{NO}_3^- / \text{Cu}^{2+} + \text{NO}$		
$\text{Mg} + \text{H}^+ / \text{Mg}^{2+} + \text{H}_2$		
$\text{MnO}_4^- + \text{Cl}^- / \text{Mn}^{2+} + \text{Cl}_2$		
$\text{MnO}_4^- + \text{Fe}^{2+} / \text{Mn}^{2+} + \text{Fe}^{3+}$		

## Experiment 8: Reactions of Hydrocarbons: Alkanes and Alkenes

### Theory

Organic compounds are compounds that contains carbon atom as part of its composition. Hydrocarbons are organic compounds containing only carbon and hydrogen in its composition.

There are 3 classes of hydrocarbons: alkanes (single bonds) such as methane, alkenes (double bonds, hexene), alkynes (1-heptyne, triple bonds). Alkanes are classified as saturated but un-reactive and the other two as unsaturated hydrocarbons and very reactive.

In this experiment, different test will be done to help us identify alkanes and alkenes.

**Apparatus:** Dropper, 4 test tubes, stop cork or rubber stopper.

**Reagents:** Hexane, Heptane, 0.5% aqueous potassium permanganate, 95% ethanol, 6m Sulfuric acid, cyclohexene

### Procedure:

#### Part A: Baeyer's Test

Add 2 mL of 95% ethanol to each of the two test tubes **A and B**. To test tube **A**, add 2 drops of hexane and none to test tube **B**. Add 10 drops of 0.5% aqueous potassium permanganate to both test tubes and then shake for 5 minutes. If any color change occurs, add extra 10 drops of the permanganate solute to both tubes and shake for 5 more minutes. Record your observation in Table 8.1. If the color of the permanganate disappears or a brown precipitate or solution is formed, then an alkane is present.

**Part B:** Alkanes do not react with sulfuric acid.

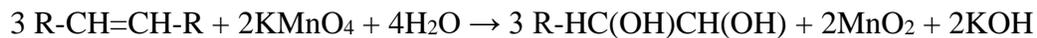
Put 2 mL of sulfuric acid in a test tube and add 10 drops of hexane. Shake very well for 2 minutes. Record if heat is evolved or hexane dissolves in Table 8.2. Repeat the procedure with heptane.

#### Part C: Alkenes

##### Baeyer's Test

In the absence of other groups that can be easily oxidized, neutral or alkaline aqueous permanganate (purple color) oxidizes alkenes to glycols with a deposit of brown manganese dioxide (brown color). The glycol is colorless and water soluble and hence this test is specific for alkenes.

### Experiment 8 Con'td



Glycol

brown

Add 2 mL of 95% ethanol to each of the two test tubes **A and B**. To test tube **A**, add 2 drops of cyclohexene and none to test tube **B**. Add 10 drops of 0.5% aqueous potassium permanganate to both test tubes and then shake for 5 minutes. If any color change occurs, add extra 10 drops of the permanganate solute to both tubes and shake for 5 more minutes. Record your observation in Table 8.3. If the color of the permanganate disappears or a brown precipitate or solution is formed, then an alkene is present

**Caution:** (1). If any permanganate spills on you, wash it off immediately with a lot of water.

Permanganate stains.

(2) Be careful with sulfuric acid. It is highly corrosive. Do not cover the test tube containing sulfuric acid with your thumb while shaking the test tube. Use a stop cork or glass stopper.

**Waste:** Do not pour waste down the drain. Use the special containers provided in the laboratory.

**Experiment 8 Cont'd.****RESULT****Name****Department****Date****Table 8.1**

<b>Procedure</b>	<b>Tube A</b>	<b>Tube B</b>
Add 2 mL of 95% Ethanol		
Add 2 drops of Hexane to <b>A</b> and <b>none to B</b>		
Add 2 mL of 0.5% aqueous permanganate to both tubes and shake for 5 minutes		
If there is a color change, add 10 drops to 0.5% aqueous permanganate and shake for 5 more minutes		

**Conclusion:****Part B****Table 8.2**

<b>Procedure</b>	<b>Observation</b>
Put 2 mL of Sulfuric acid in a test tube	
Add 2 drops Hexane , cover with a stopper and shake vigorously	
<b>Conclusion</b>	
Put 2 mL of Sulfuric acid in a test tube	
Add 2 drops Heptane, cover with a stopper and shake vigorously	
<b>Conclusion</b>	

**Experiment 8 Con'td:****RESULT****Name****Department****Date****Part C: Alkenes****Table 8.3**

<b>Procedure</b>	<b>Tube A</b>	<b>Tube B</b>
Add 2 mL of 95% Ethanol		
Add 2 drops of cyclohexene to <b>A</b> and <b>none to B</b>		
Add 2 mL of 0.5% aqueous permanganate to both tubes and shake for 5 minutes		
If there is a color change, add 10 drops to 0.5% aqueous permanganate and shake for 5 more minutes		

**Conclusion:****Question**

1. 2 drops of an unknown hydrocarbon was added to 2 mL of ethanol in a test tube. 10 drops of alkaline permanganate was added and the mixture shaken vigorously for 20 minutes. No color change or precipitate was observed. What is the name of this test method and what type of hydrocarbon is the unknown?
2. When 10 drops of aqueous permanganate was added to an unknown hydrocarbon in ethanol, a brown precipitate was noticed at the bottom of the test tube containing the mixture. Is the unknown an alkane or alkene?

## Experiment 9: Reactions of Alkynes and Aromatic Hydrocarbons

### Theory

Alkynes are straight chain unsaturated hydrocarbons with triple bonds ( $\equiv$  bonds) while aromatic compounds are cyclic unsaturated hydrocarbons. Both are very reactive and react by substitution methods. Terminal alkynes react with ammoniacal silver nitrate to form terminal acetylides and both classes of hydrocarbons react with strong oxidizing agents to form glycols.



**Apparatus:** Dropper, 4 test tubes, stop cork or rubber stopper.

**Reagents:** 1-Heptene, 2-methyl-3butyn-2-ol, aqueous ammoniacal silver nitrate, 0.5% aqueous potassium permanganate, 95% ethanol, Toulene (methyl benzene), Ammonia, dilute nitric acid.

### Procedure

#### Part A

##### Preparation of Ammoniacal Silver Nitrate

Put 5 mL of 2% silver nitrate solution in a test tube. Slowly add concentrated ammonium hydroxide (ammonia) by drops to the silver nitrate solution. A brown precipitate is formed. Continue to gently add the ammonia solution drop-wisely until the brown precipitate dissolves. Split the resulting solution into two equal halves (**A** and **B**).

##### Formation of Metallic Acetylides

Add 2 mL of water to test tube **A** and stir to mix. Then add 2-3 drops of 2-methyl-3-butyn-1-ol. Record your observation in Table 9.1.

**Caution: Acetylides are explosive when dry. Gently add dilute nitric acid to dissolve the precipitate and then gently pour the waste down the drain**

Add equal amount of 95 % ethanol to test tube **B** and then add 2-3 drops of 1-heptyne. Gently shake to mix. Record your observation in Table 9.1

**Caution: Acetylides are explosive when dry. Gently add dilute nitric acid to dissolve the precipitate and then gently pour the waste down the drain**

#### Part B: Aromatics

## Experiment 9 Con'td

### Test for Unsaturation: Baeyer's Test

In the absence of other groups that can be easily oxidized, neutral or alkaline aqueous permanganate (purple color) oxidizes aromatics to glycols with a deposit of brown manganese dioxide (brown color). The glycol is colorless and water soluble and hence this test is specific for unsaturated hydrocarbons.

Add 2 mL of 95% ethanol to each of the two test tubes **A and B**. To test tube **A**, add 2 drops of toluene and none to test tube **B**. Add 10 drops of 0.5% aqueous potassium permanganate to both test tubes and then shake for 5 minutes. If any color change occurs, add extra 10 drops of the permanganate solute to both tubes and shake for 5 more minutes. Record your observation in Table 9.2. If the color of the permanganate disappears or a brown precipitate or solution is formed, then an alkane is present

### Questions

1. When Bromine is added to a solution of 1-heptyne in a test tube, the brown color of bromine disappears. What type of reaction is this and write a balanced equation for the reaction?
2. Can hept-2-ene react with ammoniacal solution of silver nitrate.

**Experiment 9: Con'td.****RESULT****Name****Department****Date****Part A: Alkynes****Table 9.1**

<b>Test</b>	<b>Observation</b>	<b>Observation</b>
Add ammonia solution in drops to silver nitrate solution and split equally into test tube A and B		
Add 2-3 drops of 2-Methyl-3butyn-1-ol to tube A		
Add 2-3 drops of 1-heptyn-1-ol to test tube B		
Add dilute nitric acid to both tubes until precipitate dissolves. Then discard in container provided by the instructor.		

**Experiment 9: Con'td****RESULT****Part B: Aromatics****Table 9.2**

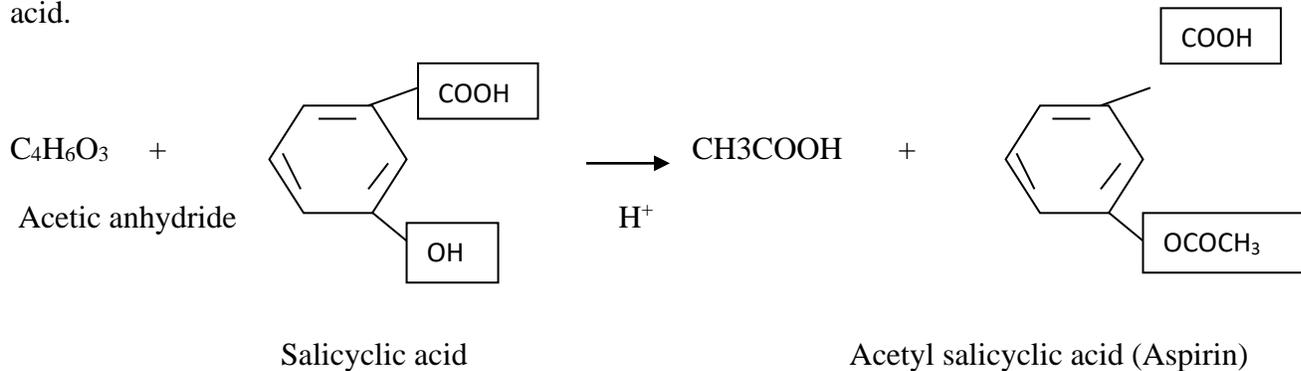
<b>Procedure</b>	<b>Tube A</b>	<b>Tube B</b>
Add 2 mL of 95% Ethanol		
Add 2 drops of cyclohexene to <b>A</b> and <b>none to B</b>		
Add 2 mL of 0.5% aqueous permanganate to both tubes and shake for 5 minutes		
If there is a color change, add 10 drops to 0.5% aqueous permanganate and shake for 5 more minutes		

**Experiment 10: Esterification Process: Preparation of Aspirin**

## Theory

Esters (R-COOR) are prepared by reacting an organic acid with an alcohol. But phenols (an aromatic alcohol) cannot be esterified by direct reaction with organic acids. For example, phenols can only be esterified by reacting it with acetic anhydride in the presence of a mineral acid such as phosphoric acid.

Aspirin (acetyl salicylic acid) is a common analgesic and anti-platelet medication which can be prepared by reacting salicylic acid (a phenol) with acetic anhydride in the presence of phosphoric acid.



**Apparatus:** Ice bath with ice, 50 mL Erlenmeyer flask, 600 mL beaker, tripod stand, wire gauze, retort stand and its clamp, Bunsen burner, thermometer, filtration assembly (Buchner flask, rubber tubing, filter paper, suction source), evaporating dish, oven, filter paper, weighing balance, melting point tube, glass rod, fume cupboard.

**Reagent:** salicylic acid, acetic anhydride, phosphoric acid, cold water,  $FeCl_3$ , methanol

## Procedure

Weigh 2g of salicylic acid (MW= 138.12 g/mol) into the 50 mL Erlenmeyer flask and add 5 mL of acetic anhydride ( $d=1.082$  g/mL, MW= 102.09 g/mol). Gently add 5 drops of 85% phosphoric acid. Clamp the neck of the flask and place in a water bath (600 mL flask with water placed on a wire gauze and a tripod stand). Heat the water bath to 75-80°C with a Bunsen burner and maintain the temperature for 15-20 minutes. Occasionally, stir the mixture with the glass rod.

Use the clamp to lift the flask from the water bath and re-attach to the retort stand. Place the flask attached to the retort stand in the hood (fume cupboard) and add 2 mL of cold water to destroy excess acetic anhydride (instructor will provide cold water). Fumes of hot acetic acid will be produced by this addition of cold water. Allow the reaction to subside and then add additional 20 mL of cold water. Place the flask in an ice bath for 10 minutes. Watch aspirin precipitate out of the mixture. Weigh a filter paper and place in the suction (filtration)

## Experiment 10 Cont'd

assemblage. Pour all the contents of the flask onto the filter paper and rinse the flask with extra cold de-ionized water. Allow the suction to run for another 5-10 minutes to further dry the aspirin. Turn the suction off and lift the filtered aspirin onto an evaporating dish and place in the oven. Dry for about 15-20 minutes in the oven. Take the dry aspirin out of the oven and allow to cool in the air. Weigh the aspirin and calculate the % yield (theoretical yield- experimental yield).

### Test for Purity

There are two methods for testing for impurity in the prepared sample of aspirin: melting point and the phenol test with  $\text{FeCl}_3$

### Melting Point Test

Prepare a melting point sample by filling a capillary tube with the aspirin crystal (2-3 m tall). Use the melting point apparatus to determine the melting point of aspirin. Set the initial temperature of the melting point apparatus to  $\leq 120^\circ\text{C}$ .

### Phenol Test For Presence of Salicyclic Acid

Fill one test tube (**A**) with few crystals of the prepared aspirin and another test tube (**B**) with few crystal of salicyclic acid. Add 5.0 mL of methanol and 1 drop of 1%  $\text{FeCl}_3$  solution. Shake and record your observation. (If salicyclic acid is present in the final mixture, a purple colored present will be formed)

**Caution: Handle the phosphoric acid and the acetic anhydride with care. Pour all waste down the drain.**

### Question

1. Salol (phenyl salicylate, is the phenyl ester of salicyclic acid), is another important pharmaceutical product made from salicyclic acid. Phenol cannot be directly esterified by direct reaction with salicyclic acid hence an indirect method must be devised. Suggest a process method that might be used to prepare Salol from phenol and salicyclic acid?
2. Calculate the theoretical yield of acetyl salicyclic acid (aspirin) if 2.5 g o salicyclic acid is reacted with excess acetic anhydride and 3 drops of phosphoric acid?

**Experiment 10 Con'td: Esterification**

Name

Department

Date

**Table 10**

#	Test	Result
1	Mass of Flask	
2	Mass of flask + salicylic acid	
3	Mass of salicylic acid	
4	Moles of salicylic acid	
5	Mass of acetic anhydride	
6	Moles of acetic anhydride	
7	Limiting reactant	
8	Theoretical yield, moles	
9	Theoretical yield, grams	
10	Mass of filter paper	
11	Mass of evaporating dish	
12	Mass of dish, paper, product	
13	Mass of product	
14	Percentage yield	
15	Melting point of product	
16	Color of FeCl <sub>3</sub>	
17	Color of positive FeCl <sub>3</sub> test	

## Experiment 11      Qualitative Analysis of Anions

### Theory

Nature is full of mixtures of many substances which are useful to mankind. For example, herbs have been found to be of medicinal use to man and are often mixtures of active and inactive ingredients. Test must be made to detect, identify, and establish presence of the active component in a herbal mixture. **This method is called qualitative analysis.**

In qualitative analysis, a specific reagent is added to the component been sort and a characteristic response for the component will be observed if the component is present. Sometimes, interference from other components might occur. In such a case, separation of the component been sort will be required before the characteristic test for the component is done. If separation is not possible, indirect method might be applied for the component to be properly identified.

In this experiment, several known solutions and unknown mixture will be tested for the presence of some anions.

**Apparatus:** Measuring cylinder, 4 test tubes, test tube rack, water bath

**Reagents:** See Appendix B for test reagents, unknown mixtures.

### Procedure

Wash the test tube with soap and rinse thoroughly with water after each use. Add 1.0 mL of the known solution to test tube **A** and 1.0 mL of the unknown solution to test tube **B**. Perform each test simultaneously on both test tubes in the order shown in Appendix B. Record your observations and confirm the components of the unknown.

### Waste:

**Place all TCE waste in the container provided by the instructor. Do not pour down the drain**

### Caution:

**Be careful with concentrated acids such as sulfuric and nitric acid. They are very corrosive and burns and dissolve books, clothing and flesh. In case of a splash on any of these items, rinse with copious amount of water. Silver nitrate also stains but it is temporary**

**Use tongs and holders to prevent burns and turn all gases before leaving the laboratory.**

### Experiment 11 Con'td:

Table 11

Name

Department

Date

Anion	Observation (Known)	Observation (Unknown)	Net Ionic Equation
$\text{CO}_3^{2-}$			
$\text{CrO}_4^{2-}$			
$\text{NO}_3^-$			
$\text{PO}_4^{3-}$			
$\text{SO}_4^{2-}$			
$\text{SCN}^-$			
$\text{I}^-$			
$\text{Br}^-$			
$\text{Cl}^-$			

### Questions.

1. An unknown solution contains either NaCl or NaCrO<sub>4</sub>. What reagents is needed to add to the unknown in order to be able to identify the anion present in the unknown?
2. Silver nitrate is added to an acidic solution containing NaCl or NaBr, producing a precipitate. What one reagent can be added to identify the compound? Indicate the possible result?

## Experiment 12 Qualitative Analysis of Cations I: The Chloride Group

### Theory

Elements form cations with specific physical and chemical characteristics which can be used to identify them in a mixture. Some of the identification process is a one step process while others may involve multiple processes. The end result is to be able to separate and identify the desired component in a mixture.

Precipitation, ionic equilibrium, acid-base properties, pH, redox reactions, and complexation are few methods available for separation and identification of cations. Chemical properties can be used to classify cations into groups and further classification within each group can be made to identify the members of each group. Group I Cations is called the Chloride group and it includes: Lead ( $\text{Pb}^{2+}$ ), mercury ( $\text{Hg}^{2+}$ ), and  $\text{Ag}^+$ . Test procedure will be used to isolate and identify each ion in a mixture.

Each of these ions can be separated from other soluble cations by precipitating them as chloride salts. Further separation into individual cations can be done as follows: (a) add hot water to the mixture of the three salts. Lead chloride will dissolve in hot water while the other two does not. So, lead will be separated by decanting the hot water solution of lead from the rest and confirmed by adding  $\text{CrO}_4$  solution to give a yellow lead chromate precipitate. Silver chloride can be separated from mercury chloride by adding aqueous ammonia solution. A  $\text{Ag}(\text{NH}_3)_2^+$  complex is formed which is soluble while the mercury-ammonia complex is insoluble (black/white precipitate (ppt)).

**Apparatus:** Test tubes, measuring cylinder, dropper, centrifuge machine, water bath,

**Reagents:** Known, unknown, 6M HCl, cold water, 6M acetic acid, 1 M  $\text{K}_2\text{CrO}_4$ , 6M  $\text{NH}_3$ , 6M  $\text{HNO}_3$ .

### Procedure

Thoroughly wash each test tube and rinse with copious amount of water before using it for another test. This is to avoid contamination and false results.

#### Part A

Place 1.0 mL of known and unknown into test tube **A** and **B**. To each of the test tube add 2 drops of 6M HCl, mix and centrifuge for about 2 minutes. Add one more drop of 6M HCl and if precipitate is formed, centrifuge again. Repeat the addition of one drop of HCl and centrifuging until no precipitates appears again (**Caution: Do not add too much acid because soluble lead and mercury chloride complexes may be formed**). Decant the liquid and keep it if groups II and III ions are present or else discard it.

## Experiment 12 Con'td:

### Test for Cations I

#### Part B: Identification of $\text{Pb}^{2+}$ Ions

Add 1 mL of cold water to the precipitate from Part A, mix and centrifuge. Decant the supernatant solution and discard. Add 1.0 mL of water to the tube, stir and heat it up in a water bath for 3 minutes. Centrifuge while hot. Decant the supernatant solution into another test tube. Add 1.0 mL of water to the ppt. and centrifuge. Decant the top solution and add it to the first one decanted previously. Save the ppt. and allow the decanted solution to cool down. Then add 1 drop of 6M acetic acid and 3 drops of 1 M  $\text{K}_2\text{CrO}_4$ . Presence of yellow ppt. confirms lead.

#### Part C Identification of $\text{Hg}_2^{2+}$ Ions

Add 10 drops of 6M  $\text{NH}_3$  to the precipitate from Part B. Stir the mixture while adding ammonia. A precipitate of black (Hg) or white ( $\text{HgNH}_2\text{Cl}$ ) confirms the presence of Hg. Decant the supernatant solution and use it in the next part.

#### Part D Identification of $\text{Ag}^+$

Add 6M  $\text{HNO}_3$  in drops to the solution from part C until the solution turns litmus paper red (acidic). Presence of a white ppt. indicates the presence of  $\text{AgCl}$ .

**Waste: Put all waste in the special container provided by the instructor**

**Caution: Be careful with all strong acids. They are corrosive. If in contact with them, wash the affected area with a lot of water.**

**Remember to turn all gas outlets off before leaving the laboratory**

### Questions

1. When HCl is added to an unknown solution containing a group 1 cation, a white ppt. was observed. Which ion may be present and write the net ionic equation for the formation of the precipitate?
2. If water is added to the ppt. from question one and heated for some minutes in a water bath a part of the ppt. is observed to dissolve. What can you say about the cations present in these unknown and what test can you use to confirm your suggestion.



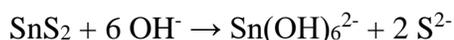
## Experiment 13: Qualitative Analysis of Cations II: The Hydrogen Sulfide Group

### Theory

The ions included in this group include:  $\text{Bi}^{3+}$ ,  $\text{Cu}^{2+}$ ,  $\text{Sn}^{4+}$ ,  $\text{Pb}^{2+}$ ,  $\text{Cu}^{2+}$ ,  $\text{As}^{3+}$ ,  $\text{Sb}^{3+}$ ,  $\text{Hg}^{2+}$  which forms precipitates with hydrogen sulfide ( $\text{H}_2\text{S}$ ) in acidic medium ( $\text{pH}=0.3-0.5$ ). The source of the sulfide for these experiments is aqueous solution of thioacetamide ( $\text{CH}_3\text{CSNH}_2$ ) which on heating gives  $\text{H}_2\text{S}$ .



$\text{Bi}^{3+}$ ,  $\text{Cu}^{2+}$ ,  $\text{Sn}^{4+}$ , will be used as representative for this group. The separation procedure for this group involves formation of the sulfide followed by separation by appropriate reagent suitable for each ion. For example, Tin (IV) sulfide is separated from the thioacetamide solution by adding sodium hydroxide which result into the formation of soluble tin hydroxo- complex.



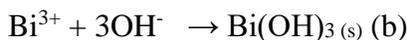
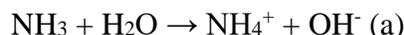
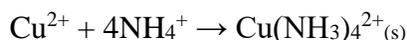
$\text{HCl}$  can be added to the decanted hydroxo-tin complex to give a yellow tin sulfide precipitate.



The Bismuth and copper sulfides can be identified by a two step reaction. The first step is to dissolve both sulfides with nitric acid.



and then add aqueous ammonia solution. Bismuth gives a white precipitate while copper gives a deep blue copper complex. The process for bismuth complex formation is thought to be a two step process indicated as equations **a** and **b**.



**Apparatus:** Test tubes, test tube holder, dropper, litmus paper, centrifuge, water bath

**Reagent:**  $\text{Bi}^{3+}$ ,  $\text{Cu}^{2+}$ ,  $\text{Sn}^{4+}$ , 6M  $\text{NH}_3$ , 6M  $\text{HCl}$ , 6M  $\text{HNO}_3$ , 6M  $\text{NaOH}$ , 1M thioacetamide (TAA)

## Experiment 13 Con'td

### Procedure

#### Part A

Measure 1.0 mL of known (all the 3 cations in the group) and 1.0 mL of unknown solution and put in test tube **A** and **B** respectively. Add 6M HCl or 6M NH<sub>3</sub> in drops to the test solutions and test with litmus paper until the pH is within 0.3-0.5. When the pH is within the desired range, then add 12-15 drops of thioacetamide (TAA) to each solution. Stir the solutions while heating in a water bath for the next 5 minutes. Centrifuge for about 2 minutes. Add 2-3 drops of TAA to check for complete precipitation and allow to stand for about 1 minute. If precipitate appears, heat in water bath for 2 -3 more minutes and centrifuge. Decant and discard the supernatant liquid. Add 10-15 drops of 1M NH<sub>4</sub>Cl to the precipitate. Stir, warm, centrifuge, decant and discard the supernatant liquid

#### Part B

Add 1.0 mL of 6M NaOH to the precipitate from part A. Stir and heat with water bath for about 2 minutes. Centrifuge and decant the top solution in another test tube. Keep the precipitate. Add in drops, 6M HCl to the decanted solution and test with litmus paper until the solution is acidic (red litmus color). Then add 4-6 drops of 1M TAA. Heat and stir for about 5 minutes. A yellow precipitate of tin may be formed

#### Part C

To the precipitate from part B, add 2 mL of 6M HNO<sub>3</sub>. Heat and stir for about 1 minute. Centrifuge the solution if any insoluble sulfur remains in the mixture. Decant the top solution and add 6M NH<sub>3</sub> until it is basic to litmus (blue color). Bi<sup>3+</sup> is confirmed if a white precipitate is formed or a deep coloration for Cu<sup>2+</sup>.

**Waste:** Flush all waste down the drain with a lot of water

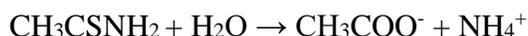
**Caution:** Be careful with the concentrated acids, and bases. All H<sub>2</sub>S test should be done in the hood. Turn all gases outlet off before leaving the laboratory



## Experiment 14 Qualitative Analysis of Cations III: Ammonium Sulfide Group

### Theory

The ions in this group include:  $\text{Cr}^{3+}$ ,  $\text{Co}^{2+}$ ,  $\text{Zn}^{2+}$ ,  $\text{Fe}^{2+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Al}^{3+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Mn}^{2+}$ . They are called the **ammonium** sulfide group which forms precipitates as hydroxides or as sulfides in alkaline solution saturated with hydrogen sulfide ( $\text{H}_2\text{S}$ ) (from thioacetamide (TAA)). The alkaline condition is from ammonium acetate formed by hydrolysis of thioacetamide and ammonium



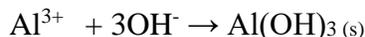
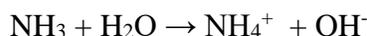
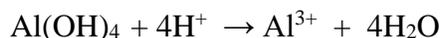
Acetamide                      acetate

chloride, which is added as a buffer to keep the solution basic. The acetamide and the acetate ions affect the pH of the solution by hydrolysis and the sulfide ion produces hydroxide ions when it precipitates metal sulfides.

In this experiment, will concentrate on the separation of the following ions:  $\text{Cr}^{3+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Al}^{3+}$ ,  $\text{Ni}^{2+}$ . Analysis of this group of ions is done after all interfering ions from cations in groups I and II have been removed and identified. Separation is done by starting with addition of NaOH to the solution. A reddish-brown precipitate of ferric hydroxide ( $\text{Fe}(\text{OH})_3$ ) will be formed and the nickel hydroxide ( $\text{Ni}(\text{OH})_2$ ) will be formed as a green precipitate. If excess NaOH is used, a soluble aluminum complex ( $\text{Al}(\text{OH})_4^-$ ) will be formed. On addition of NaOCl,  $\text{Cr}^{3+}$  (dark blue) will be oxidized to  $\text{CrO}_4^{2-}$  (yellow).  $\text{Ni}^{2+}$  and  $\text{Fe}^{2+}$  do not easily form hydroxides neither are they affected by the hypochlorite and therefore remain insoluble.



The soluble hydroxo complexes of this group such as  $\text{Al}(\text{OH})_4^-$ , are precipitated by adding acid to the solution (removes the hydroxo complexes) followed by addition of aqueous ammonia. The reaction results into formation of white gelatinous precipitate of  $\text{Al}(\text{OH})_3$ .



The remaining ion in solution is the chromate ion which can be precipitated by barium chloride to form a yellow precipitate of barium chromate.

The nickel (II) and Fe(III) insoluble hydroxide complexes are separated from the solution by adding an acid. Subsequent addition of alkaline aqueous ammonia solution results into the

## Experiment 14 Con'td.

precipitation of the iron as hydroxides while nickel remain in solution as blue amino complex ( $\text{Ni}(\text{NH}_3)_6^{2+}$ ).

Presence of the iron can be confirmed by adding an acid and potassium thiocyanate to the complex. A red solution of  $\text{Fe}(\text{SCN})_2^{2+}$  is formed which confirms the presence of iron. Similarly, presence of Ni is confirmed by forming a red precipitate when reacted with dimethylglyoxime ( $\text{HC}_4\text{H}_7\text{N}_2\text{O}_2$ ).

**Apparatus:** Large test tubes, dropper, water bath, centrifuge

**Reagents:**  $\text{Cr}^{3+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Al}^{3+}$ ,  $\text{Ni}^{2+}$ , 6M NaOH, 6M HCl, 6M  $\text{HNO}_3$ , 6M  $\text{NH}_3$ , 0.5M NaOCl, dimethylglyoxime ( $\text{HC}_4\text{H}_7\text{N}_2\text{O}_2$ ), 0.1% Aluminon (ammonium salt of aurin tricarboxylic acid), 6M Acetic acid, 0.1M  $\text{BaCl}_2$ ,  $\text{NH}_4\text{Cl}$ , 0.15M KSCN

### Procedure

#### Part A: Initial Separation into Subgroups

Put 1.0 mL of the unknown mixture and the known mixture into large test tube **A** and **B** respectively. Add 1mL of NaOH to both test tubes and gently boil for 1 minute. Maintain the initial volume by adding water to the solution while boiling. Add 1.0 mL of 0.5M NaOCl and gently boil for another minute. Remove from heat and then add 8 drops of 6M  $\text{NH}_3$  and boil for another minute. Remove from the heat and transfer the contents of both test tubes to a centrifuge test tube. Centrifuge for about a minute and decant the supernatant solutions from the precipitate. Keep the liquid and precipitates for further analysis.

#### Part B: Analysis of Aluminum Ion

Add 6M of  $\text{HNO}_3$  in drops to the solutions from part A until the solutions becomes acidic to litmus. Then add 6M  $\text{NH}_3$  also in drops to turn the solution basic with litmus. The result is a white gelatinous precipitate of  $\text{Al}(\text{OH})_3$ . Centrifuge for about a minute and decant the supernatant solution. Keep the solution for the next step(part C). To confirm the presence of Aluminum, add 1 mL of hot water to the precipitate, centrifuge and decant the supernatant solution (discard). Then add 4 drops of 6M  $\text{HNO}_3$  to the precipitate. This will dissolve the precipitate. Finally, add 2-3 drops of 0.1% Aluminon to the solution followed by drops of 6M  $\text{NH}_3$  until the solution turns basic. The result is a red globular precipitate of  $\text{Al}^{3+}$ .

#### Part C: Determination of $\text{Cr}^{3+}$ Ion

Add 2-3 drops of 6M acetic acid to the solution from part B. A yellow precipitate of barium chromate is formed when 4-6 drops of barium chloride is added to the acidified solution.

## **Experiment 14 Con'td:**

### **Part D: Isolation of Ni<sup>2+</sup> Ion**

Retrieve the precipitate from part A and add 1.0 mL of 6M HNO<sub>3</sub> and heat in a water bath until all the precipitate dissolves. Allow to cool and add 8-10 drops of 1M NH<sub>4</sub>Cl, followed by drops of 6M NH<sub>3</sub> until it turns basic with litmus. Addition of about 8-10 drops of 6M NH<sub>3</sub> will re-precipitate iron as hydroxides. Keep the precipitate for the next part (part E). Centrifuge and decant the supernatant solution. Add 3-5 drops of 1% dimethylglyoxime to the decanted solution. Appearance of a red precipitate of Ni<sup>2+</sup> ion confirms its presence.

### **Part E: Detection of Fe<sup>3+</sup>**

To the precipitate from part D, add 8-10 drops of 6M HCl. Stir and add 2-3mL of water and 2-3 drops of 0.15M KSCN. Reaction results into the formation of a red solution of FeSCN<sup>2+</sup>.

**Waste:** Chromium waste should be put in the container provided by the instructor. Other wastes can be flushed down the drain.

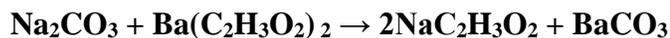
**Caution:** Be careful with all the strong acids and ammonia. Turn off all the gas valves before leaving the laboratory. **Keep safe by acting safe**



## Experiment 15: Limiting Reactants

### Theory

The stoichiometry of any reaction determines the maximum amount of products that such reaction will produce. For example, in the equation shown below,



one mole of sodium carbonate reacts with one mole of barium acetate (**reactants**) to produce two moles of sodium acetate and one mole of barium carbonate (**products**). If in the beginning, half mole of sodium carbonate was used, then all of it will be consumed and one mole of sodium acetate will be produced and similarly if half mole of barium acetate was present in the beginning, then half mole of barium carbonate will be produced. In summary, we can say that the amount of product produced will be determined by the amount of **reactants** in short supply at the beginning of the reaction. The reactants in short supply at the beginning of the reaction is called the **limiting reactant**.

This experiment is going to use a mixture (unknown amounts) of sodium carbonate and barium acetate which will be dissolved in water. The two reactants will dissolve in water and produce the product shown in the equation above. The amount of barium carbonate produced will be determined by the amount of the limiting reactant present in the unknown mixture. By weighing the precipitate, one will be able to determine the amount of the limiting reactants present in the unknown mixture. One can also determine the amount of the excess reactant by adding more of the limiting reactant followed by separation, drying and weighing.

**Apparatus.** Rubber tubing's, Buchner funnel and flask, filter paper, water tap with suction point, dessicator, oven, evaporating dish.

**Reagent.** Barium acetate, sodium carbonate, water.

### Procedure

Weigh approximately 1.5 g ( $\pm 0.01$  g) of unknown sample into a previously weighed 250 mL beaker. Add 100 mL of water and stir for about two minutes dissolves. Put the appropriate pre-weighed filter paper in a Buchner funnel, insert the funnel into a rubber bung and then insert the funnel with the rubber bung into a separation flask. Attach rubber tubing to the flask and to the suction part of the tap. Allow the tap to run smoothly to create suction. Wet the filter paper with water, stir the beaker and gently transfer all the contents of the beaker into the funnel. Rinse the beaker with water to make sure all the solids are transferred to the funnel. If the filtrate is cloudy, pour it back into the beaker and re-filter using the same filter paper. Repeat the procedure until a clear filtrate is obtained. Weigh a dry evaporating dish, Transfer the filter paper with the solid to the evaporating dish. Dry the filter with the solid in an oven for about 15-20 minutes. Place the

## Experiment 15 Con'td

evaporating dish in a dessicator and allow to cool. Weigh the cooled solid and calculate the amounts of reactants.

Divide the filtrate equally into two measuring cylinder. Add several drops of 1M barium acetate to one cylinder to test for excess sodium carbonate. If a precipitate is formed, then sodium carbonate is the excess reactant and barium acetate is the limiting reactant. Test the second cylinder with 1 M sodium carbonate to determine which is the limiting reactant and the excess reactant. These two tests will help to determine the limiting and excess reactants in the unknown.

Add 5 ml of the solution that produce the precipitate in the test above to the portion of the filtrate that produced the precipitate. Use another paper to filter the precipitate. Transfer the precipitate into the oven and dry for 15-20 minutes. Cool the precipitate in a dessicator for and weigh the precipitate. Record all your results in Table 1.

**ATTENTION: All chemical wastes may be flushed down the drain. Use tongs at all times to hold the evaporating dish.**

### Questions

1. Does Line 23 add to the same amount of the starting material of the unknown? Why or why not?
2. A 16 g of hydrogen is reacted with 16 g of Oxygen. What is the limiting reactant and how many grams of water is produced?  $2\text{H}_2 + \text{O}_2 \rightarrow 2\text{H}_2\text{O}$
3. When solutions of barium chloride and sodium chromate are mixed, a yellow precipitate of barium chromate is formed. Describe in general terms how you would determine experimentally the identity of the limiting reactant?

**Experiment 15 Con'td****RESULT****Name****Department****Date****Table 15**

#	Test	Result
1	Mass of beaker	
2	Mass of beaker + unknown	
3	Mass of unknown	
4	Mass of filter paper #1	
5	Mass of evaporating dish #1	
6	Mass of dish, paper, BaCO <sub>3</sub>	
7	Mass of Ba CO <sub>3</sub>	
8	Moles of BaCO <sub>3</sub>	
9	Does addition of Ba(C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> ) <sub>2</sub> to filtrate result in a precipitate?	
10	Does addition of NaCO <sub>3</sub> to filtrate result in a precipitate?	
11	What is the limiting reactant?	
12	Mass of filter paper #2	
13	Mass of evaporating dish #2	
14	Mass of dish, paper and precipitate 2	
15	Mass of precipitate 2	
16	Moles of precipitate 2	
17	Total moles of BaCO <sub>3</sub> (line 8 + (2Xline 16))	
18	Total mole of limiting reactant	
19	Total moles of excess reactant	
20	Ration of excess reactant to limiting reactant	
21	Total mass of limiting reactant	
22	Total mass of excess reactant	
23	Total mass of all reactants	
24		

## Experiment 16: Molecular Weight of A Volatile Liquid

### Theory

For an ideal gas at room temperature, the Pressure, volume and temperature of a gas or a volatile liquid obey the law

$$2. \quad PV = nRT$$

n is the number of moles, R the gas constant, T is temperature in Kelvin.

If the number of moles (n) = weight/molecular weight (m/MW), then,

$$PV = nRT/MW$$

Hence,

$$MW = nRT/PV$$

If the mass and volume of a gas can be measured at a known temperature and pressure, then its molecular weight can be determined

**Apparatus:** 1x 125 mL Florence flask (round bottom flask), 600 ml beaker, aluminum foil, rubber band, tripod stand, burner, pin, balance, thermometer.

**Reagents:** Unknown

**Procedure.** Weigh the clean Florence flask, rubber band and the foil. Put approximately 5.0 mL of the unknown in the flask and cover with the foil. Tie the foil with the rubber band and hold the flask with a retort stand. Gently place 600 mL beaker on a tripod stand and fill it with warm water. Gradually lower the flask into the beaker as far as it can go. Use the pin to make tiny holes on the foil. Heat the beaker with a low flame (unknown is flammable) until the water boils. Continue to heat the beaker until all the unknown liquid vaporizes and none is left in the flask. Measure the temperature of the water. Turn of the burner and lift the flask out of the beaker. Cool the flask with cold water until it is at room temperature. Weigh the flask. Repeat the same procedure one more time.

At the end of the second run, fill the flask with water, replace the foil and rubber band and then weigh the flask. Using the this weight and the density of water, calculate the volume of the flask. The volume of the flask is the same as the volume of the vapor of the unknown.

**Note:** The pressure of the vapor in the flask equalized with the atmospheric pressure during cooling. Hence, the atmospheric pressure in the laboratory will be used as the pressure of the gas.

**Waste Disposal:** Return any unused waste to the laboratory instructor.

**Experiment 16****RESULT**

Name

Department

Date

**Table 16**

	Procedure	Test1	Test 2
1	Mass of flask, foil and band		
2	Mass of flask, foil, band + Vapor		
3	Mass of vapor (Line 2-1)		
4	Temperature of Vapor		
5	Atmospheric Pressure		
6	Mass of water filled flask, foil, band		
7	Mass of water in flask		
8	Volume of flask (Vapor)		
9	Molecular Weight of Unknown		
10	Average Molecular Weight		

3.

**Questions**

1. At  $99^{\circ}\text{C}$  and 750 torr, the vapor filling a 125.0 mL flask weighs 0.320 g. Calculate the molecular weight using the ideal gas law.
2. Would you expect your experimental result for the molecular weight to be higher or lower than the true value if:
  - a) There is an impurity in the unknown? Explain
  - b) There is water remaining on the exterior of the flask when it is weighed while containing the water? Explain

## Experiment 17: Chromatographic Separation Methods

### Theory

Nature abounds with mixtures that need to be separated into its components for the use of man. For example, many pharmaceutical products (natural or synthetic) are initially mixtures which are subjected to separation techniques for isolation and identification of its active component and impurities. Separation techniques include solvent extraction, ion pairing or exchange, liquid-liquid extraction, gas phase, solid- liquid extraction and chromatographic extractions.

This experiment is about chromatographic method of separation of a group of indicators. Chromatography involves partitioning mixtures between a solid phase (stationary phase) and gas or liquid phase (mobile phase). The separation process involves constant absorption and desorption of the components of the mixture between the mobile phase and the stationary phase. This is an equilibrium process that depends on the relative solubilities of the components of the mixture in the mobile phase or its affinity for the stationary phase. The more soluble a component is in the mobile phase, the faster it will be eluted or transported along the stationary phase. If the resolution is good enough, each component will appear as individual fractions that can be collected and identified by a detector.

In paper chromatography, paper is the stationary phase while different solvents such as methanol, acetonitrile etc. are used as mobile phase to elute components of a mixture. Components migrate at different rate depending on their relative affinity for the surface of the paper and the solvent. At a specific condition, the distance travelled by a components (solute) is a constant percentage of the distance travelled by the solvent (solvent front). The constant percentage is called,  $R_f$  and is good enough to identify that particular solute.

$$R_f = \text{distance traveled by the solute/distance traveled by solvent}$$

**Apparatus:** Chromatographic paper, depression plate, pencil, ruler, rubber band, developing chamber or clear glass jar, hood, stapler, micropipette.

**Reagents:** Indicators, unknown mixture, acetone, n-butanol, ammonia

**Procedure:** Add two drops of each of the known indicator and the unknown mixture into three different spots on the depression plate. Put 25 mL of each of butanol and ammonia into the developing chamber or jar and cover with a lid. Place the developing chamber in the hood. Pour another 25 mL of ammonia into another jar or developing chamber, seal it with a lid and also place it in the hood.

Handle the chromatographic paper at the edges only. Finger prints can interfere with the movement of the solvent. Draw a light pencil line 1.5 cm from and parallel to the edge of the shorter or longer side of the chromatographic paper. Spot the first known sample with a micro

## Experiment 17 Con'td

pipette on the pencil line and allow to dry. Repeat the spotting three more times. Clean the micropipette with acetone and allow it to dry. Using a pencil, label the spot **A**. Repeat the process for the second unknown and the mixture leaving at least 2.5 cm in between each sample. Label the second known mixture as **B** and the unknown as **C** and allow the spots to dry.

Roll the paper to form a cylinder with a little gap between the edges and staple. Gently lower the paper into the developing chamber with the side containing the spots at the bottom of the jar or developing chamber. Close the developing chamber (jar) and allow the chromatogram to develop for one hour.

After one hour, remove the paper from the chamber and immediately use your ruler and pencil to mark the solvent front. Expose the paper to the ammonia chamber. Identify the spot(s) for each sample and mark the center of each spot. Measure the distance travelled by each spot and the solvent front. Spray the paper with water at the solvent front and expose to ammonia again. Mark and measure the distance travelled if any new spot(s) is found.

Calculate the  $R_f$  value for each component found in the chromatogram.

**Experiment 17****RESULT**

Name

Department

Date

**Table 10**

Indicator	Color of Spot in Ammonia	Distance from origin to solvent front	Distance from Origin to center of Spot	R <sub>f</sub>
Unknown				

**Questions.**

1. A spot and its solvent front migrated 6.80 cm and 10.50 cm from the origin respectively. Calculate the spot **R<sub>f</sub>**?
2. What is the physical property applicable to separations with paper chromatography?
3. If ink is used to mark the origin, how would the chromatogram look?

## Experiment 18: Equilibria and Le Chatelier's Principle

### Theory

In a chemical reaction, reactants are consumed to make products. In some cases, all the reactants are consumed to make the product (reactions go to completion)



and in some cases, not all the reactants are consumed to make the product (partial reaction).



Another example of a partial reaction is the production of hydrogen iodide (at room temperature) shown in the equation below. In such reactions, the rate of the forward reaction (product formation) and the rate of the



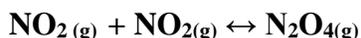
backward reaction (reactant formation) will be the same after some time. That stage is called a **dynamic equilibrium** state. If the ratio of the concentration of the product with that of the reactant is calculated, the ratio will give a constant value at that given condition (temperature, concentration, pressure, heat of reaction etc). That ratio is called the equilibrium constant ( $K_{\text{eq}}$ )

$$K_{\text{eq}} = \frac{[\text{HI}]^2}{[\text{H}_2][\text{I}_2]} \quad (4)$$

### Le Chatelier's Principle

Le Chatelier's principle talks about how equilibrium responds to changes (stress) that happens to a system at equilibrium. He stated that the effect of any changes made to a system at equilibrium is annulled (cancelled) by the equilibrium re-establishing itself or the equilibrium position will shift so as to minimize the effect of such changes. For example, in forming **HI** (eqn. 3), if the concentration of **H<sub>2</sub> is increased** after equilibrium is established, equilibrium will respond by consuming more of **I<sub>2</sub> in the same proportion to produce more of HI**, hence equilibrium will be re-established.

Another example is nitrogen dioxide gas which dimerizes to form dinitrogen dioxide in an exothermic reaction process



If extra heat is added to the system, the backward reaction, which is endothermic, will be favored and equilibrium will shift towards the left (reactants, nitrogen dioxide) to re-establish equilibrium. The converse is also true for this reaction

## Experiment 18 Con'td

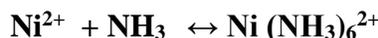
**Apparatus:** 3 test tubes, plastic droppers, brush

**Reagents:** Water, 0.1 M NiCl<sub>2</sub>, 6M NH<sub>3</sub>, 6M HCl, 1M MgCl<sub>2</sub>, 1M MgCO<sub>3</sub>, 1M NH<sub>4</sub>Cl, 0.2% phenolphthalein, 6M NaOH, 0.1% bromocresol

### Procedure

#### Part A. Complex Ion Formation With A Metal

Rinse a test tube with water. Add 1 ml of 0.1 M NiCl<sub>2</sub> solution to the test tube. Record the color in Table 11a. Add 2 drops of 6M NH<sub>3</sub> solution the test tube and record your observation. Finally add 2 drops of 6M HCl and note the color. Explain your observations with Le Charteliers principle



#### Part B: Alteration of Equilibrium with A Common Ion

Equilibrium can be altered by adding an ion that is common to the reactant or the product. This is called the Common ion Effect. A common ion is used to dissolve the precipitate of a sparingly soluble salt.

- (i) Put 10 drops of 1M MgCO<sub>3</sub> in a clean test tube. Then add 5 drops of 1M MgCl<sub>2</sub>. Record your observation in Table 11b and explain the reaction with the principle of common ion effect and Le Charteliers principle



- (ii) Add 5 drops of 1M MgCl<sub>2</sub> to a clean test tube. Put 2 drops of 6M NH<sub>3</sub> to the solution in the test tube to produce a gel-like precipitate. Then add 5 drops of 1M NH<sub>4</sub>Cl and shake the mixture very well.



Observe what happens and record it in Table 11c. Explain your observation using common ion effect and Le Charteliers principle.

**Experiment 18 Con'td****RESULT**

Name

Department

Date

**Table 18a**

<b>Reaction</b>	<b>Observation</b>
Color of Ni <sup>2+</sup>	
Color of Complex	
Color after adding HCl	

(b) Explain your observations with Le Chatelier's principle

**Table 18b**

<b>Reaction</b>	<b>Observation/Net Ionic Reaction</b>
10 drops of 1M MgCO <sub>3</sub> in a test tube	
10 drops of 1M MgCl <sub>2</sub> to the test tube	

(c) Explain your observations using common ion effect and Le Chatelier's principle

**Experiment 18 Con'td****RESULT****Name****Department****Date****Table 11c**

<b>Reaction</b>	<b>Observation/Net Ionic Reaction</b>
5 drops of 1M MgCl <sub>2</sub> in a test tube	
2drops of 6M NH <sub>3</sub> to the test tube	$\text{MgCl}_2 + \text{NH}_3 + 2\text{H}_2\text{O} \leftrightarrow \text{Mg}(\text{OH})_2 + \text{NH}_4^+$
Add 5 drops of 1M NH <sub>4</sub> Cl and mix well	

(d) Explain your observations using common ion effect and Le Chatelier's principle

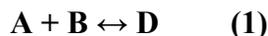
**Question.**

1. A reaction is already at equilibrium and was subsequently heated. Can you predict how equilibrium will respond? If not, what other information would you need to be able to predict which direction equilibrium will shift?

## Experiment 19: Estimating Equilibrium Constant

### Theory

Nature is filled with many different types of reactions. Some reactions occur at very slow rate (nature making fossil fuel), some at moderate rate (rusting of nails), and some at a very fast rate (explosions). Many of these reactions go to completion and some do not. For the reactions that do not go to completion (eqn. 1), equilibrium will be established at which the rate of formation of the product (forward reaction) will be the same as the rate of re- forming the reactants (the backward reaction). All equilibrium is established at a specific condition such as temperature and pressure (for gaseous system).



The equilibrium constant for equation 1 is written as

$$\mathbf{K_e = [Product]/[Reactant] = [D]/[A][B]}$$

It is easy to estimate  $\mathbf{K_e}$  since the initial concentration of the reactants and the equilibrium concentration of the reactants and product can be calculated. For example, if the initial concentration of  $\mathbf{A}$  and  $\mathbf{B}$  is  $\mathbf{M}$  moles and that of the product is  $\mathbf{x}$  moles at equilibrium, then the equilibrium concentration of  $\mathbf{A}$  and  $\mathbf{B}$  will be  $(\mathbf{M-x})$  moles. Therefore,

$$\mathbf{K_e = [x]/[M-x][M-x]}$$

There are many ways in which we can measure the equilibrium concentration of the product. One of such ways is using Ultraviolet- Visible spectrometry (UV-Vis) if the product is colored. In UV-Vis, the amount of light absorbed the product is proportional to the concentration of the product as expressed in Beer-Lamberts law.

$$\mathbf{A = abc}$$

**A= Absorbance, a = molar absorptivity, b= cell length, c= molar concentration**

To be able to determine the concentration of the product, a calibration curve must be obtained with the UV-Vis spectrometer using known concentration of the product. A plot of the absorbance of the product at different concentrations gives a straight line curve which can be used to estimate the unknown concentration of the product. The slope of the curve is **ab** and can be used to calculate the molar absorptivity (**a**) of the product.

## Experiment 19 Con'td

### Procedure

#### Part A: Calibration Curve

Make a 10 mL stock solution of Ferrous Thiocyanate ( $[\text{FeSCN}]^{2+}$ ) by adding 5.0 mL of 0.2 M  $\text{Fe}(\text{NO}_3)_3$ , 1.0 mL of  $\text{HNO}_3$ , and 4.0 mL of water in a 20 mL volumetric flask. Mix thoroughly with a glass rod. The concentration of  $\text{Fe}^{3+}$  is much greater than that of the  $\text{SCN}^-$ , hence it is assumed that equilibrium will favor product formation and all the  $\text{SCN}^-$  is consumed.

Calibration standards are prepared with the following dilution

1. 5 mL of stock solution + 5 mL of water
2. 4 mL of stock solution + 6 mL of water
3. 2 mL of stock solution + 8 mL of water
4. 1.0 mL of stock solution + 9 mL of water

Measure the absorbance or transmittance of the calibration standards ( $A = 2 - \log T$ ) at 447 nm. 0.002 M  $\text{Fe}(\text{NO}_3)_3$  can be used as the reference standard. Absorbance is plotted against the concentration to obtain a calibration curve.

#### Part B: Estimating Equilibrium Constant

Prepare a stock solution for the estimation of the equilibrium constant as follows: 35 mL of 0.002 M  $\text{Fe}(\text{NO}_3)_3$  in 1 M of  $\text{HNO}_3$  and 20 mL of 0.002 M KSCN. Then prepare the following concentration from this stock solution

Solution #	0.002M $\text{Fe}(\text{NO}_3)_3$ (mL)	0.002M KSCN (mL)	$\text{H}_2\text{O}$ (mL)
5	5	1	4
6	5	2	3
7	5	3	2
8	5	4	1
9	5	5	0

### **Experiment 19 Con'td**

Mix the mixtures thoroughly and measure its absorbance or transmittance and that of the reference solution at 447 nm.

**Waste: All waste can be flushed down the drain**

**Experiment 19 Con'td:****RESULT****Name****Department****Date****Part A: Calibration Curve****Table 19.1****Solution #**

[FeSCN <sup>2+</sup> ]	1	2	3	4
% T at 447nm				
Absorbance				

**Table 19.2****Part B: Estimating the Equilibrium Constant**

Solution #

	5	6	7	8	9
Initial mol Fe(NO <sub>3</sub> ) <sub>3</sub>					
Initial mol KSCN					



$$K_e = \frac{[\text{FeSCN}^{2+}]}{[\text{Fe}^{3+}][\text{SCN}^-]}$$

**Experiment 19 Con'td:****RESULT**

Name

Department

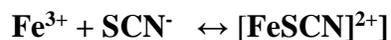
Date

**Table 19.3****At Equilibrium**

	5	6	7	8	9
%T					
Absorbance					
[FeSCN <sup>2+</sup> ] from graph					
Mol FeSCN <sup>2+</sup>					
Mol SCN <sup>-</sup>					
Mol Fe <sup>3+</sup>					
[SCN <sup>-</sup> ]					
K <sub>e</sub>					

Average K<sub>e</sub> =**Questions**

1. Calculate the initial number of moles of a Fe<sup>3+</sup> and SCN<sup>-</sup> prepared from 0.004M Fe(NO<sub>3</sub>)<sub>3</sub> and 0.006M of KSCN?
2. Convert 50% T to absorbance
3. Calculate the equilibrium constant (K<sub>e</sub>) for the reaction shown below



If the initial [Fe<sup>3+</sup>] = 3x 10<sup>-2</sup>mmol and [SCN<sup>-</sup>] = 1 x 10<sup>-4</sup>mmol and the equilibrium amount of [FeSCN]<sup>2+</sup>] = 2 x 10<sup>-5</sup> mmol?

## Experiment 20: Determination of pH of Solutions

### Theory

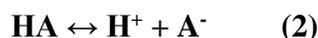
Arrhenius defined an acid as a substance that produces hydrogen ions in solution ( $\text{H}^+$ ) and a base as one that produces hydroxyl ions in solution ( $\text{OH}^-$ ). Acidity and alkalinity (basicity) is a characteristic of all solutions that needs to be determined or measured. Determining the molar concentration of  $\text{H}^+$  or  $\text{OH}^-$  ions give us the degree of acidity or alkalinity of that solution. The degree of acidity is expressed as the pH of that solution and it is defined as

$$\text{pH} = -\log[\text{H}^+] \quad (1)$$

The dissociation constant of pure water,  $K_w = [\text{H}^+][\text{OH}^-] = 10^{-14}$  at room temperature

It's  $[\text{H}^+] = [\text{OH}^-] = 10^{-7}$  and therefore its  $\text{pH} = 7$  which indicates that pure water is a neutral solution.

The extent of the dissociation of an acid in water is a measure of its acidity and it can be measured as a function of its dissociation constant  $K_a$



$$K_a = \frac{[\text{H}^+][\text{A}^-]}{[\text{HA}]} \quad (3)$$

Some acids have large dissociation constants and are completely dissociated in solution (hydrochloric acid). Others are partially dissociated in solution and are considered to be weak acid (acetic acid). If we solve for  $[\text{H}^+]$  in eqn. 3,

$$[\text{H}^+] = K_a \cdot \frac{[\text{A}^-]}{[\text{HA}]}$$

$$\text{pH} = \text{p}K_a + \log \frac{[\text{A}^-]}{[\text{HA}]} - \text{Henderson-Hasselbalch equation}$$

The importance of pH is demonstrated in **Buffer** solutions. Buffer solutions could be an acid or a basic buffer solution that resist changes in pH when small amounts of acid or base is added to it. Usually, a buffer is the weak acid and its salt or a weak base and its salt (e.g. acetic acid/sodium acetate). If a small acid is added to the buffer, it reacts with the salt to form the weak acid hence there is no change in pH. This same thing is observed when a small base is added to a basic buffer solution.

**Apparatus:** Weighing balance, 2x 250 mL beaker, measuring cylinder, 250 mL conical flask, burette, pH meter, 2 x 100 ml beakers

**Reagents:** Water, unknown weak acid 0.2M NaOH, phenolphthalein, 0.1M HCl, 0.1M NaOH.

## Experiment 20 Con'td

### Procedure

Weigh 1.50 g of unknown weak acid into the 250-mL beaker and dissolve with 100 mL of water. Divide the solution into two equal halves with a measuring cylinder. Put one half in a 250 mL conical flask, rinse the measuring cylinder and pour the rinse into the sample in the conical flask. Add 2-3 drops of phenolphthalein indicator and titrate the solution with 0.2 M NaOH to the end point. Then add the other half of the acid to the neutral solution and mix thoroughly. This final solution has equal amount of the acid and its salts. Hence,  $\text{pH} = \text{pK}_a$ . Use the pH meter to determine the pH of this mixture. Repeat the procedure one more time and find the average pH.

**Experiment 20: Con'td****RESULT**

Name

Department

Date

**Table 13**

Data	First Attempt	Second Attempt
pH of Final solution		
pK <sub>a</sub> of unknown		

(b) Average pK<sub>a</sub> =**Questions:**

1. Calculate the K<sub>a</sub> for nitrous acid in solution containing 0.01M HNO<sub>2</sub> and 0.01M NaNO<sub>2</sub> and pH = 3.5?
2. A liter of solution with pH 7 that contains 0.1M of an acid (pK<sub>a</sub> = 6.8) is required to be prepared. Is the amount of the salt needed greater or less than 0.1M?
3. Calculate the pH of a buffer containing 4.0 g of acetic acid (MW = 60.0 g/mol, K<sub>a</sub> = 1.74 x 10<sup>-5</sup>) and 2.8 g of sodium acetate (MW = 82.0 g/mol) in 1 L of solution?

## Experiment 21: Chemical Kinetics

### Theory

The kinetic theory indicates that when two atoms or molecules colliding with sufficient energy (> activation energy of reaction) a reaction occurs to form a new substance. How fast or slow (**rates of reaction**) the reaction is depends on many factors such as temperature, concentration of reactants, presence of a catalyst, polarity, bond energies etc. example of a slow reaction is the rusting of nails. It is a natural process that takes about 3-5 days to occur if the conditions are right. A fast reaction is exemplified by the explosions of bombs or by dissolving a carbonate with an acid to give bubbles. We are going to monitor the effect of temperature and concentration on reaction rates in this experiment.

For a reaction such as



The rate of reaction at constant temperature is measured as the rate of appearance of the product or the rate of disappearance of the reactants:

$$R = k_1[A]^y[B]^x = k_2[C]^m[D]^n$$

Where  $k$  = rate constant,  $x$  and  $y$ ,  $m$  and  $n$  are the order with respect to A and B

The overall order of reaction =  $y + x$  or  $m + n$  and are usually positive integers, 0, 1, 2 and/or 3.

### Effect of Concentration on Rate of Reaction

How does the rate order affect the rate of reaction? In equation 1, if the concentration of B is doubled and that of A is held constant, the rate will be doubled if the reaction is a first order reaction (i.e.  $b=1$ ). If the reaction is a second order reaction ( $b=2$ ) and the same change is made, the rate will be quadrupled (increase 4 times).

### Effect of Temperature on Rate of Reaction

When temperature is increased, the rate of reaction will increase. The converse is also true. The Arrhenius equation states that

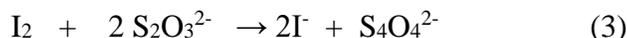
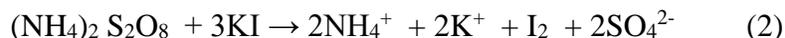
$$\log k = -E_a/2.303RT + C$$

where  $k$  is the rate constant,  $R$  = gas constant (8.314 J/mol),  $T$  = temperature in Kelvins,  $C$  = specific reaction constants,  $E_a$  = activation Energy

## Experiment 21: Con'td

The reaction of a peroxodisulfate ( $S_2O_8$ ) with an Iodide ( $I^-$ ) in the presence of starch will be used to measure the rate of reaction. The reaction is a two-step reaction as indicated in the equation shown below:

### Experiment 21 Con'td



### Iodine thiosulfate

Reaction 2 produces iodine that forms a blue black color with starch, while reaction 3 delays formation of iodine so that the reaction rate may be measured. The rate of reaction is expressed as:

$$R = k[S_2O_8]^x[I]^{-y}$$

### Determination of the Order of Reaction (x and y)

To determine the order of reaction, the temperature and the concentrations of  $K^+$  and  $NH_4^+$  will be kept constant while the concentrations of  $I^-$  and  $S_2O_8^{2-}$  will be varied. The time of reaction will be measured and the reaction rate ( $r^1$ ) calculated with the equation

$$r^1 = 1000/\text{reaction time}$$

(set up a simultaneous equations for two  $r^1$  ones to solve for x and y)

### Activation Energy ( $E_a$ )

To determine the activation energy, the concentrations of the reactants are kept constant while the temperature is varied. Using Arrhenius equation, plotting  $\log k$  vs.  $1/T$  will give a slope that is equal to  $-E_a/2.303R$  and an intercept of C.  $E_a$  is then calculated from the slope of the curve.  $\log k$  is directly proportional to the rate  $r^1$ , hence  $r^1$  will be plotted against  $1/T$  to obtain the slope.

**Apparatus:** Timers, thermometers, water baths,

**Reagents:** 0.1M  $(NH_4)_2S_2O_8$ , 0.1M  $(NH_4)_2SO_4$ , 0.2 M KCl, 0.2M KI, 0.005 M  $Na_2S_2O_3$  3% Starch

## Experiment 21 Con'td

### Procedure

The following factors affect the results obtained in this experiment: age of solution, temperature and cleanliness of glassware.

### Part A: Determination of the reaction Order

Prepare the solutions for this experiment as indicated in the table below. Prepare each reaction mixture in two separate flasks. Pour the two contents of one flask into the other and mix by

### Experiment 21 Con'td

swirling the flask and start the timer at the same time. Measure the reaction time for the blue-black color of starch to develop. Record the temperature of the final solution for reaction 1.

**Table 21**

**Reaction Mixture**

	1 (mL)	2 (mL)	3 (mL)
<b>Flask 1</b>			
KI	20	10	20
KCl	0	10	0
Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	10	10	10
3% Starch	4 drops	4 drops	4 drops
<b>Flask II</b>			
(NH <sub>4</sub> ) <sub>2</sub> S <sub>2</sub> O <sub>8</sub>	20	20	10
(NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub>	0	0	10

## Experiment 21 Con'td

### Part B: Determination of the Activation Energy

Reaction one concentrations will be used for this determination. Three rate of reactions at different temperatures will be measured. One determination has been made from part A. The other two determinations will be made at 40°C and in ice. Flask 1 and II will be prepared with

the same concentrations of reaction 1. The two flasks will be placed in a water bath until their temperature reaches 40°C. Then the contents of both flasks will be mixed together and the time of appearance of the blue-black color of starch. The process is repeated but the two flask placed in an ice bath until the temperatures of the two flask are the same. Then the contents are mixed together and the reaction time noted. Note the temperatures in all cases.

### Calculations

For reaction mixtures 1 and 2

$$r^1/r^2 = k^1[I^-]_1^x[S_2O_8^{2-}]_1^y/k^1[I^-]_2^x[S_2O_8^{2-}]_2^y$$

$$k^1 = k^1 \text{ and } [S_2O_8^{2-}]_1^y = [S_2O_8^{2-}]_2^y$$

$$\text{then } r^1/r^2 = [I^-]_1^x/[I^-]_2^x$$

$$x = \log (r^1/r^2)/\log([I^-]_1^x/[I^-]_2)$$

using reaction 1 and 3

$$y = \log (r^1/r^3)/\log([S_2O_8^{2-}]_1/[S_2O_8^{2-}]_3) \text{ where } ([I^-]_1 = [I^-]_3)$$

**Waste:** All waste can go down the drain

**Cautions:** Do not use thermometers to stir the solution

### Experiment 21 Con'td

Name \_\_\_\_\_

Department \_\_\_\_\_

Date \_\_\_\_\_

#### Part A

##### Reaction Mixture

	1	2	3
Reaction Time			
Relative rate, $r^1$			
Initial Conc., $I^-$			
Initial Conc., $S_2O_8^{2-}$			

Order of reaction with respect to  $I^-$  \_\_\_\_\_ (round to nearest integer)

Order of reaction with respect to  $S_2O_8^{2-}$  \_\_\_\_\_ (round to nearest integer)

#### Part B

##### Reaction Mixture

	1	4	5
Reaction Time			
Relative rate, $r^1$			
Temperature			
Log $r^1$			
1/T			

Slope of line \_\_\_\_\_ (attach graph)

Activation Energy \_\_\_\_\_

## APPENDIX A

Table 7.3 Table of Solubilities of Some Compounds

Anion	Soluble Except	Insoluble Except
Acetate, $C_2O_4^-$	All are soluble	All are soluble
Arsenate, $AsO_4^{3-}$		$NH_4$ , K, Na
Bromide, $Br^-$	Pb, Hg, Ag	
Carbonate, $CO_3^{2-}$		$NH_4$ , K, Na
Chlorate, $ClO_4^-$	All are soluble	
Chloride, $Cl^-$	Pb (cold water), Hg, Ag	
Chromate, $CrO_4^{2-}$		$NH_4$ , K, Na
Fluoride, $F^-$	Al, Ba, Ca, Fe(III), Pb, Mg, Sr	
Hydroxide, $OH^-$		$NH_4$ , K, Na
Iodide, $I^-$	Pb, Hg, Ag	
Nitrate, $NO_3^-$	All are soluble	
Nitrite, $NO_2^-$		$NH_4$ , K, Na
Oxalate, $C_2O_4^{2-}$		$NH_4$ , K, Na
Oxide, $O^{2-}$		Ba, Ca, K, Na, Sr
Pechlorate, $ClO_4^-$	K	
Phosphate, $PO_4^{3-}$		$NH_4$ , K, Na
Silicate, $SiO_4^-$		$NH_4$ , K, Na
Sulfate, $SO_4^{2-}$	Ba, Ca, Pb, Hg, Ag, Sr	
Sulfide, $S^{2-}$		$NH_4$ , K, Na, Ba, Ca
Sulfite, $SO_3^{2-}$		$NH_4$ , K, Na
Thiocyanate, $SCN^-$	Hg, Ag	

## Appendix B: Test for Anions

#	Anion	Test
1	Carbonate, $\text{CO}_3^{2-}$	Add 1 mL of 6M HCl to 1.0 mL of $\text{Na}_2\text{CO}_3$ solution or unknown. Evolution of $\text{CO}_2$ gas (colorless, odorless bubbles) is a positive test for a carbonate
2	Chromate, $\text{CrO}_4^{2-}$	Add 5 drops of 0.1M $\text{AgNO}_3$ to 1.0 mL of $\text{K}_2\text{CrO}_4$ or unknown. Presence of red precipitate of $\text{AgCrO}_4$ is a positive test for chromate
3	Nitrate, $\text{NO}_3^-$  Brown Ring test	Add 5 drops of 0.5M $\text{FeSO}_4$ to 1.0 mL of $\text{NH}_4\text{NO}_3$ or unknown. Mix well. Slant the test tube and gently add 1 mL of 18M $\text{H}_2\text{SO}_4$ down the side of the tube until a layer is formed at the bottom of the tube. Gently place the test tube in a vertical position and leave in the test tube rack for some minutes. A brown ring of $\text{Fe}(\text{NO})^{2+}$ is formed at the interface between the two layers for a positive test for nitrates
4	Phosphate, $\text{PO}_4^{3-}$	Add 1.0 mL of 6M $\text{HNO}_3$ and 1.0 mL of $(\text{NH}_4)_2\text{MoO}_4$ to 1.0 mL of 0.1M $\text{Na}_3\text{PO}_4$ and 1.0 mL of the unknown, then heat in a water bath for some few minutes. A yellow precipitate of $(\text{NH}_4)_3\text{PO}_4 \cdot 12\text{MoO}_3$ is a positive test for phosphate ion
5	Sulfate, $\text{SO}_4^{2-}$	Add 1.0 mL of 6M HCl and 5 drops of 0.1M $\text{BaCl}_2$ to 1.0 mL of 0.5M $\text{Na}_2\text{SO}_4$ and 1.0 mL of the unknown. Presence of white powdery-like precipitate confirms a sulfate ion
6	Thiocyanate, $\text{SCN}^-$	Add 1.0 mL of 6M acetic acid to 1.0 mL of known and unknown solution. Mix very well and then add 1.0 mL of 0.1M $\text{Fe}(\text{NO}_3)_3$ . Presence of a deep red complex of $\text{FeSCN}^{2+}$ confirms the Thiocyanate ion.
7	Iodide, I <sup>-</sup>	Add 5 drops each of acetic acid and 1,1,1-trichloroethane (TCE) to 1.0 mL of 0.1M NaI and the unknown and mix well. Add 2 drops of 0.5M NaOCl and mix again. Iodide is oxidized to Iodine to give a lavender color in TCE.
8	Bromide without Iodide present, $\text{Br}^-$	Add 5 drops each of acetic acid and 1,1,1-trichloroethane (TCE) to 1.0 mL of 0.1M NaBr and the unknown and mix well. Add 2 drops of 0.5M NaOCl and mix again. Bromide is oxidized to Bromine to give an orange color in TCE

### Appendix B Con'td: Test for Anions

#	Anion	Test
9	Bromide with Iodide present, Br <sup>-</sup>	Add 5 drops each of acetic acid and 1,1,1-trichloroethane (TCE) to 1.0 mL of 0.1M NaBr and the unknown and mix well. Add 2 drops of 0.5M NaOCl and mix again. The iodide is oxidized to Iodine and solution will turn lavender in TCE. Continue adding NaOCl until the water layer turns colorless. Bromide is oxidized to Bromine to give an orange color in TCE
10	Chloride without SCN <sup>-</sup> , I <sup>-</sup> , Br <sup>-</sup> present, Cl <sup>-</sup>	Add 1.0 mL of 6M HNO <sub>3</sub> and 3 drops of 0.1M AgNO <sub>3</sub> to 1.0 mL of 0.1M of NaCl and unknown. Presence of a white precipitate of AgCl confirms Cl <sup>-</sup> ion
11	Chloride with SCN <sup>-</sup> , without I <sup>-</sup> , Br <sup>-</sup> present, Cl <sup>-</sup>	SCN <sup>-</sup> forms white precipitate with AgNO <sub>3</sub> and hence must be removed before testing for the chloride ion. Add 1.0mL of 6M HNO <sub>3</sub> to 1 mL of unknown in and evaporating dish. Heat until volume is reduced to half. Allow to cool and then pour into a clean test tube. Then add 1.0 mL of 6M HNO <sub>3</sub> and 3 drops of 0.1M AgNO <sub>3</sub> . Presence of a white precipitate of AgCl confirms Cl <sup>-</sup> ion
12	Chloride with SCN <sup>-</sup> , I <sup>-</sup> , and/or Br <sup>-</sup> present, Cl <sup>-</sup>	If SCN <sup>-</sup> is present proceed as in Test 11. If not add 1.0 mL of 6M HNO <sub>3</sub> and 3 drops of 0.1M AgNO <sub>3</sub> . A yellow precipitate of AgI and/or AgBr masks the presence of AgCl and must be removed. To remove the interference, add 6M NH <sub>3</sub> until the solution turns litmus blue (basic). (Silver iodide and bromide are insoluble while the chloride is soluble). Centrifuge and decant the supernatant solution. Add 1.0 mL of 6M HNO <sub>3</sub> will give a white precipitate of AgCl.

### **More Readings**

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3. Wismer, R.K;Qualitative Analysis with Ionic Equilibrium. Third Edition. 1991. Macmillan Publishing Company. New York. USA.