

**LABORATORY MANUAL**  
**FOR**  
**INORGANIC CHEMISTRY**  
**FIRST EDITION**

**DEPARTMENT OF CHEMISTRY**  
**BABCOCK UNIVERSITY. ILISAN. REMO. OGUN STATE**

**2014**

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## **Policies, Practices, and Procedures**

### **SAFETY**

The most important events in the laboratory are safety and the right to know the physical and chemical hazards present in the laboratory. Follow instructions as stated in the laboratory manual and be careful with acids and bases.

**!!!PLEASE DO NOT DISOBEY LABORATORY RULES. IT MIGHT PROVE FATAL!!!!**

### **PROTECTION**

1. Instructors, laboratory technologist, laboratory assistants, students and other personnel that work in the laboratory must wear approved goggles at all times when in the laboratory. It is a recommendation not to wear contact lenses when in working in the laboratory
2. It is advisable to wear old clothes that cover head to toe when working in the laboratory. Long sleeve shirts and long pants are recommended. Shorts, midriff tops and sandals are not allowed.
3. Long hair is to be tied and well tucked away. It is a fire hazard when let loose or flying
4. Students are not allowed to work in the laboratory alone
5. Horseplay or other pranks are prohibited in the laboratory
6. Smoking, eating, drinking or applying makeup is not allowed in the laboratory

### **LABORATORY MAINTENANCE**

1. Make sure your Laboratory space(s) is cleaned. Also clean all the equipments and returned them to their assigned positions. Failure to do so will result to a zero grade for the experiment. NO exceptions please.
2. All glassware must be cleaned before it is put away.
3. Use sponges to clean bench tops and wiping of non-hazardous materials.
4. Laboratory instructors are the ONLY one allowed to clean up corrosive or toxic materials
5. Sweep up broken glassware with a broom and collect with the dust pan and then place in the special container provided for glasses.
6. No debris of any type should be left in the sink. Put all debris in allocated containers
7. Make sure all drawers are properly closed and locked when necessary.

## **GENERAL INFORMATION**

1. Dispense organic solvents, strong acids and bases and other volatile solvents in the fume hoods.
2. **No fee will be collected for broken equipment or glassware. Each broken glassware and equipments will be replaced with two of similar type by the culprit.**

## **LABORATORY TECHNIQUES**

1. Use proper utensils such as crucible tongs to hold or move hot items.
2. Make sure there are no flammable materials near you when lightning a burner
3. Add boiling chips to liquids before heating them up. This will help to prevent bumping or boil over.
4. Place test tubes in a slanting position away from yourself and others when heating liquids. Heat liquids at the surface of the liquid.
5. Do not heat up a closed system
6. Heat all substances that emit noxious fumes under the hood
7. Use funnel to transfer liquids into a narrow neck container
8. Use a bulb or pump to pipette a liquid. Never use your mouth
9. Avoid smelling anything unless instructed to do so. While sniffing, gently waft the material towards your nose when allowed to do so
10. Do not return excess reagent to its original container
11. Do not experiment with the chemicals in the laboratory except those that your are scheduled to do.
12. Do not use your pipette or spatula to remove samples from the stock container. Use the one provided by the laboratory technologist
13. Correctly label test tubes or other containers indicating their contents
14. Strong acids and bases should be added to water and not vice versa.

## **EMERGENCIES AND FIRES**

1. Laboratory instructors are in-charge of all emergencies. Follow instructions as directed

2. All laboratory users should learn how to locate the following materials: safety shower, eyewash, blankets, fire extinguishers, first aid kit, fire alarm
3. Laboratory users should notify the laboratory instructors of any fire.
4. Turn off all gas jets if it is the source of the fire
5. All laboratory users should learn how to use the fire extinguisher

### **ACCIDENTS AND INJURIES**

1. The chemistry department does not treat injuries or illness. Any injury or illness will be referred to the University Medical Center, Ilishan, Ogun State.
2. It is the responsibility of the laboratory instructor(s) on duty to prevent further injury by taking the appropriate action after the incident. Arrangement should be made to immediately transport the victim to the Medical Center. If the injury is minor and the student can walk to the Medical Center, such student should be accompanied by another person to the Medical Center.
3. An accident report form must be filled at all times even when the victim declines Medical treatment.

### **SPECIAL WASTE**

1. The laboratory will provide label containers for hazardous waste. Read the label very well and dispose the waste appropriately.
2. At no time should organic or toxic wastes such as mercury, lead, chromium be dumped down the drain.
3. Ask when in doubt about proper disposal of waste.

### **Experiment 1: Precipitation Reactions**

## Theory

Many methods are used in determining the end points in precipitation reactions. For example, Mohr titration reactions are employed in the determination of chlorides and bromides. In the titration, a neutral chloride solution is titrated with a silver nitrate using potassium chromate as an indicator. At the end point, the chromate ions combine with the silver ions to form sparingly soluble red silver chromate. In this experiment, we have two sparingly soluble salts being silver chloride ( $K_{sol} = 1.2 \times 10^{-10}$ ) and silver chromate ( $K_{sol} = 1.7 \times 10^{-12}$ ) respectively. If for example, 0.1 M sodium chloride in the presence of dilute potassium chromate is titrated with 0.1 M silver nitrate, silver chloride is a less soluble salt and again the chloride concentration is high, hence silver chloride will be precipitated. At the first appearance of the red silver nitrate, we shall have both salts in equilibrium in solution hence;

$$[Ag^+][Cl^-] = K_{solAgCl} = 1.2 \times 10^{-10} \text{ and } [Ag^+][CrO_4^{2-}] = K_{solAgCrO_4} = 1.7 \times 10^{-12}$$

$$[Ag^+] = K_{solAgCl}/[Cl^-] = \{K_{solAgCrO_4}/[CrO_4^{2-}]\}^{1/2}$$

$$[Cl^-]/[CrO_4^{2-}] = K_{solAgCl}/K_{solAgCrO_4} = 1.2 \times 10^{-10}/1.7 \times 10^{-12} = 9.2 \times 10^{-5}$$

$$\text{At equivalence point } [Cl^-] = \{K_{solAgCl}\}^{1/2} = 1.1 \times 10^{-5}$$

$$[CrO_4^{2-}] = \{[Cl^-]/9.2 \times 10^{-5}\} = \{1.1 \times 10^{-5}/9.2 \times 10^{-5}\} = 1.4 \times 10^{-5}$$

If a more dilute potassium chromate solution is used as an indicator, it makes detection of the silver chromate more difficult (end point). This will introduce an error which can be corrected by doing a blank determination. The blank will help us to determine the volume of silver nitrate required to give a perceptible coloration when added to distilled water containing the same amount of indicator. This volume is subtracted from the volume of the standard solution used.

**Apparatus:** Weighing balance, filter paper, 2x 100 and 2x 500 ml graduated flask, stirrer, 3x250mL flask, 25mL pipette, 1.0 mL pipette, indicator dropper

**Reagents:** Pure Silver nitrate, Sodium Chloride, potassium chromate, potassium dichromate, calcium carbonate

## Procedure

### Section A: Preparation of 0.1M Silver Nitrate Standard Solution

Dry some finely powdered silver nitrate at 120°C for 2 hours. Allow to cool down and accurately weigh out approximately 8.5g of silver nitrate and dissolve in 100 mL of water in a 500ml graduated flask. Make up the solution to the mark and stir the solution thoroughly.

### Section B: Preparation of 0.1M Sodium Chloride Standard Solution

Weigh out 2.922 g of pure and dry sodium chloride and dissolve in a 100 ml flask in a 500 mL graduated flask. Dilute to the mark and stir the solution thoroughly for proper mixing.

### Section C: Preparation of the Potassium Chromate Indicator Standard Solution

4.2 g of potassium chromate and 0.7g of potassium dichromate are dissolved with water in a 100ml graduated flask. Dilute to the mark and stir very well for proper mixing

#### **Section D: The Mohr Titration (Titration of the Silver Nitrate Solution)**

Pipette 25 mL of the standard sodium chloride solution into a 250 mL conical flask resting on a white tile and add 1.0 mL of the indicator with the 1.0 mL pipette. Pour the silver nitrate into a burette and gently add to the sodium chloride solution, swirling continuously until a red color formed by the addition of each drop of the silver nitrate begins to disappear slowly. Continue the addition drop wisely until a faint but distinct color change occurs. The faint reddish brown color should persist after a brief shaking. This is the end point. If the end point is overstepped, production of a deep reddish-brown color) add more chloride solution and titrate again. Subtract the blank volume from the titration volume (this should not be more than 0.03-0.10 mL) before calculation. Repeat the titration two more times.

#### **Section E: The indicator Blank Correction**

Add 0.5 g of calcium carbonate to water that have the same volume of the volume obtained in the silver nitrate titration. Add 1.0 mL of the indicator to the blank solution. Gradually add 0.01M solution of silver nitrate to the solution until an inert white precipitate similar to that obtained in the titration of chlorides

**Experiment 1**

**RESULT**

**Name**

**Department**

**Date**

**Table 1**

	<b>Volume 1</b>	<b>Volume 2</b>	<b>Volume 3</b>	
<b>Ag(NO<sub>3</sub>)</b>				
<b>[Cl<sup>-</sup>]</b>				
<b>[CrO<sub>4</sub>]</b>				
<b>[Ag<sup>+</sup>]</b>				

### **Question**

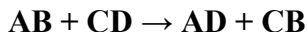
1. Why must be reaction carried out in a neutral solution?
2. What reaction will happen if the reaction is carried out in an acid solution?
3. What might happen if the solution is markedly alkaline?

## **Experiment 2: Redox and Other Chemical Reactions**

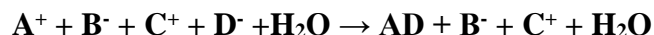
### **Theory**



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;

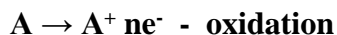


Some of the ions may appear in the same form on both sides of the equation and therefore, omitted from the net ionic equations as shown below



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 2.1

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>

**Experiment 2 Cont'd**

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>
- 6M HCl + 6M NaOH
8. 0.1M NiCl<sub>2</sub> + 1M Na<sub>2</sub>CO<sub>3</sub>

**Use Table A 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 2.2

### **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 2**

**RESULT**

**Name**

**Department**

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**Table 2.1**

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

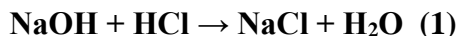
**Experiment 2 Cont'd****Table 2.2**

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 3: Equilibria and Le Chateliers 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



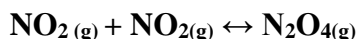
backward reaction (reactant formation) will be the same after some time. That stage is called a **dynamic equilibrium** state. If the ratio of the concentration of the product with that of the reactant is calculated, the ratio will give a constant value at that given condition (temperature, concentration, 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 Charterliers Principle

Le Charteliers 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 3 Cont'd

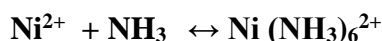
**Apparatus:** 3 test tubes, plastic droppers, brush

**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



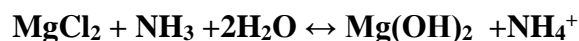
### Part B: Alteration of Equilibrium with a Common Ion

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 3 Cont'd

RESULT

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**Table 3a**

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

(b) Explain your observations with Le Charteliers principle

**Table 3b**

Reaction	Observation/Net Ionic Reaction
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 Charteliers principle

**Experiment 3 Cont'd**

**RESULT**

Name

Department

Date

**Table 11c**

Reaction	Observation/Net Ionic Reaction
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 Charteliers principle

### Question

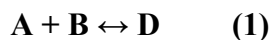
1. A reaction is already at equilibrium and was subsequently heated. Can you predict how equilibrium will respond? If not, what other information would you need to be able to predict which direction equilibrium will shift?

## Experiment 4: 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 by 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 4 Cont'd**

### **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 4 Cont'd

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 4 Cont'd:**

**RESULT**

**Name**

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**Part A: Calibration Curve**

**Table 19.1**

**Solution #**

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

**Table 4.2**

**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 4 Cont'd:**

**RESULT**

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**Table 4.3**

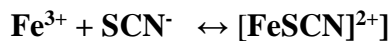
**At Equilibrium**

	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 5: Gravimetry: Determination of Oxalates**

## Theory

Gravimetric analysis is also known as quantitative analysis by weight is a process of isolating and weighing of an element or a definite compound of the element in as pure form as possible. The element or compound is separated from a weighed portion of the substance being examined. A significant part of the process involves of the element to a pure stable compound suitable for weighing. The weight of the element is then calculated from knowledge of the formula of the stable compound and the atomic weights of the constituting element. The separation of the element may be done in a number of ways that includes (i) precipitation (ii) volatilization (c) electroanalytical (d) extraction and chromatography.

A demonstration of this method will be exemplified by the determination of oxalates as calcium oxalate or calcium carbonate. Oxalates are important in medicine because of their involvement in the occurrence of the disease called kidney or gall bladder stones. The neutral solution of alkali oxalates can be acidified, heated to boiling and precipitated with boiling calcium chloride solution. The precipitate can be allowed to stand for 12 hours, filtered, washed with hot water dried and weighed as calcium oxalate or calcium carbonate or oxide.

**Apparatus:** Weighing balance, 2x 250 mL conical flask, measuring cylinder, filter paper stirring rod or spatula, filtering crucible.

**Reagents:** Hot and cold water, calcium chloride, ammonium chloride, ethanol, anhydrous diethyl ether, Bunsen burner or a heating mantle

## Procedure

Weigh 2.0 g of calcium chloride and 0.5 g of ammonium chloride. Dissolve the ammonium chloride in 50 mL of cold deionized water and the calcium chloride in 50 mL of cold water. Heat the calcium chloride solution to boiling and gradually add to the ammonium chloride solution. Allow the mixture to cool down and treat with one third of its volume of 90 percent ethanol. Allow the mixture to stand for 30 minutes. Pour the mixture through a filtering crucible fitted with a filter paper and wash the precipitate with warm water (50-60°C) until the chloride reaction is negative. Again wash the precipitate with cold water (20 mL) one time, five times with ethanol and several times with small volumes (5 mL) of dry diethyl ether. The precipitate is air dried for about ten minutes and allowed to cool down in a desiccator for another ten minutes. The precipitate is then weighed as calcium oxalate monohydrate ( $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$ ) or oven dried until a constant weight is obtained and weighed as the carbonate or oxide.

## Experiment 5: Cont'd

## RESULT

Name

Department

Date

**Table 5**

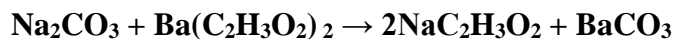
<b>Data</b>	<b>First Attempt</b>	<b>Second Attempt</b>
Initial weight of Calcium Chloride		
Final weight of the Oxalate or carbonate of oxide		
% of oxalate ion ppt.		

**Questions:**

1. Calculate the percentage of oxalate ion in 5.0 g calcium oxalate
2. What happens when an oxalate ion like calcium oxalate is heated?
3. Use an equation to explain the reaction in question 2?

## 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 6 Cont'd

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?



Name

Department

Date

**Table 6**

#	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		

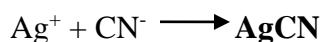
**Experiment 7: Complex Ion Formation**

## Theory

Addition of potassium cyanide (KCN) solution to a solution of silver nitrate (AgNO<sub>3</sub>) will produce a white precipitate of silver cyanide (AgCN). The solubility product of silver cyanide is exceeded for this reaction to occur



The overall reaction can be written as



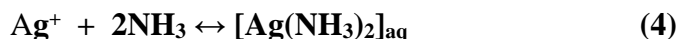
and the solubility product can also be written as

$$K_{\text{SP}} = [\text{Ag}^+][\text{CN}^-] \quad (2)$$

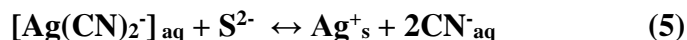
If extra amount of the KCN or other anions is added, the precipitate will dissolve to form a silver ion complex or complexes of other ions ([Ag(CN)<sub>2</sub>] or [Ag(X)<sub>n</sub>] where x = other anions).



One can conclude that a **complex** ion can be formed when an ion unites with other ions of opposite charge or neutral molecules (NH<sub>3</sub>)



If a sulfide solution is added, the complex ion can dissociate into silver ions which is then precipitated as silver sulfide



The dissociation constant for complex ion (eqn. 5) is

$$K_{\text{diss}} = [\text{Ag}^+][\text{CN}^-]^2 / [\text{Ag}(\text{CN})_2^-] \quad (6)$$

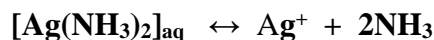
**K<sub>diss</sub> for this reaction is 1.0 x 10<sup>-21</sup> mol<sup>2</sup>/dm<sup>6</sup> at room temperature**

The value of the **K<sub>diss</sub>** is very small and it shows that the [Ag<sup>+</sup>] is very small ([CN<sup>-</sup>] is very large) that the solubility product of AgCN is not met.

The inverse of **K<sub>diss</sub>** is called **stability or formation constant** and is expressed as:

$$K = 1 / K_{\text{diss}} = [\text{Ag}(\text{CN})_2^-] / [\text{Ag}^+][\text{CN}^-]^2 = 10^{21} \text{ dm}^6/\text{mol}^2$$

The stability of complex ions varies widely among different complexing agents and is quantitatively expressed as the **stability constant**.



$$K_{\text{diss}} = [\text{Ag}^+][\text{NH}_3]^2 / [\text{Ag}(\text{NH}_3)_2]_{\text{aq}} = 6.8 \times 10^{-2} \text{ mol}^2/\text{dm}^6 \text{ and}$$

$$K = 1/K_{\text{diss}} = 1.5 \times 10^7 \text{ dm}^6/\text{mol}^2$$

The more stable a complex the larger the value of its **stability constant**

**Apparatus:** 4x 100 mL beakers, 1x 10 mL measuring cylinder, 3 x 50 mL bakere, stirrer, stopper, 4 test tubes

**Reagent:** 0.01M KCN, 0.2 M AgNO<sub>3</sub>, 0.01M sulfide salt,

### Procedure

Obtain 15ml of 0.01M KCN and 20 mL 0.2M AgNO<sub>3</sub> and 20mL of 0.01M sulfide salt into 3 x100 mL beakers. Prepare the solutions as shown in Table 7.1 into 3 test tubes. Shake each solution at intervals of 5 minutes for fifteen minutes and leave to stand for precipitation to occur. This will ensure that equilibrium is reached.

**Table 7.1**

Solution #	Volume of 0.01M KCN (mL)	Volume of 0.2 M AgNO <sub>3</sub> (mL)	Concentration (mmoles)
1	5.0	3.0	
2	5.0	4.0	
3	5.0	5.0	

Determine the concentrations of solutions 1, 2, and 3 and record it Table 7.1

$$[\text{CN}^-] = (\text{volume of 0.01 M KCN})(0.02\text{M AgNO}_3)/\text{Total volume of solution}$$

### Preparing the Calibration Curve

Decant the supernatant solution in solutions 1-3. Centrifuge the decanted solution for 2 minutes

### Experiment 7 Cont'd

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

### Further Reading

**LABORATORY MANUAL**  
**FOR**  
**ORGANIC CHEMISTRY**  
**FIRST EDITION**

**DEPARTMENT OF CHEMISTRY**  
**BABCOCK UNIVERSITY. ILISAN. REMO. OGUN STATE**

**2014**

**Further Readings**

1. Quantitative Skoog and West. 3<sup>rd</sup> Edition. USA
2. General Chemistry Manual. Delaware University Newark Delaware. USA
3. Vogel's Textbook of Practical Inorganic Chemistry (5th Edition) 5th Edition

## Appendix A

**Table A Table of Solubilities of Some Compounds**

<b>Anion</b>	<b>Soluble Except</b>	<b>Insoluble Except</b>
Acetate, C <sub>2</sub> O <sub>4</sub> <sup>-</sup>	All are soluble	All are soluble
Arsenate, AsO <sub>4</sub> <sup>3-</sup>		NH <sub>4</sub> , K, Na
Bromide, Br <sup>-</sup>	Pb, Hg, Ag	
Carbonate, CO <sub>3</sub> <sup>2-</sup>		NH <sub>4</sub> , K, Na
Chlorate, ClO <sub>4</sub> <sup>-</sup>	All are soluble	
Chloride, Cl <sup>-</sup>	Pb (cold water), Hg, Ag	
Chromate, CrO <sub>4</sub> <sup>2-</sup>		NH <sub>4</sub> , K, Na
Fluoride, F <sup>-</sup>	Al, Ba, Ca, Fe(III), Pb, Mg, Sr	
Hydroxide, OH <sup>-</sup>		NH <sub>4</sub> , K, Na
Iodide, I <sup>-</sup>	Pb, Hg, Ag	
Nitrate, NO <sub>3</sub> <sup>-</sup>	All are soluble	
Nitrite, NO <sub>2</sub> <sup>-</sup>		NH <sub>4</sub> , K, Na
Oxalate, C <sub>2</sub> O <sub>4</sub> <sup>2-</sup>		NH <sub>4</sub> , K, Na
Oxide, O <sup>2-</sup>		Ba, Ca, K, Na, Sr
Pechlorate, ClO <sub>4</sub> <sup>-</sup>	K	
Phosphate, PO <sub>4</sub> <sup>3-</sup>		NH <sub>4</sub> , K, Na

Silicate, $\text{SiO}_4^-$		$\text{NH}_4$ , K, Na
Sulfate, $\text{SO}_4^{2-}$	Ba, Ca, Pb, Hg, Ag, Sr	
Sulfide, $\text{S}^{2-}$		$\text{NH}_4$ , K, Na, Ba, Ca
Sulfite, $\text{SO}_3^{2-}$		$\text{NH}_4$ , K, Na
Thiocyanate, $\text{SCN}^-$	Hg, Ag	