ENGINEERING

CHEMISTRY

Practical Part

For

ENGINEERING AND APPLIED SCIENCE

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by

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Contents

Introduction

Your practical work may be composed of four different types of activities:

1. Activities where you learn practical basic skills in order to master them or where you conduct simple procedures to make observations that help illustrate concepts.
2. Investigations, often called experiments, where you will follow a complete set of directions in order to determine a set of observations or results that you will analyze in order to help you answer a question or gain a deep understanding of the development or application of a concept.
3. Exercises where you may be given a problem to solve a hypothesis in the laboratory and need to be able to decide on the proper equipment, material and procedures to perform in order to achieve the result.
4. Practical examination where you will have to demonstrate your ability in any of the above three types of activities by yourself. Remember that even if you are working in individual, paired or group, every member of the group is responsible for being able to perform every task in the activity or investigation. It is critical that the work is shared, and jobs are rotated so that every student has a chance to practice his or her skills.

General Chemistry Safety and Laboratory Rules

Chemistry laboratories can be hazardous if the rules are not followed. During a chemistry course a student may handle materials which are carcinogenic, poisonous, flammable, and explosive. Some of these materials and equipment may also cause severe burns, cuts, or bruises if handled improperly or carelessly. Most accidents that occur in the chemistry laboratory are a result of carelessness, impatience, improper or unauthorized experimentation, and disregard for safety rules or proper operating procedures. In order to minimize the chances of an accident in the laboratory certain rules and regulations must be obeyed at all times when one is working or observing in a chemical laboratory. Therefore, it is not advisable for anyone to work in a laboratory without proper knowledge of the dangers involved. Due to the inherent dangers present in a chemical laboratory exercise, it should be understood that the following rules must be obeyed to minimize the chance of an accident. The student is expected to exercise proper judgment and extreme caution at all times when working in the laboratory.

Learn and observe the safety and laboratory rules !

1. DO NOT perform unauthorized experiments or work in a laboratory alone.
2. Approved eye protection must be worn at all times in the laboratory. Tennessee State law requires the use of such devices. Eye protection must be splash proof chemical goggles and be approved by your instructor. If you do get a chemical in your eye rinse immediately with large quantities of water using the eye-wash stations.
3. Long hair and loose clothing must be confined while in a laboratory.

4. Appropriate clothing must be worn at all times while in the laboratory. Your legs must be completely covered below the knee by your choice of clothing. If your clothing does not meet the requirement you may choose to wear an approved laboratory coat or apron which does cover your legs to your knees.
5. Closed shoes with socks must be worn at ALL times open toed shoes, backless shoes, sling backs, clogs, and sandals are not permitted.
6. Know the location and proper use of fire extinguishers, fire blankets, safety showers, eye wash devices, and first aid kits.
7. Before obtaining any chemicals carefully read the label on the reagent bottles.
8. Eating, smoking, and drinking are **not** allowed in a chemistry laboratory.
9. Thoroughly wash your hands after leaving the laboratory.
10. Use the fume hoods when toxic or irritating vapors are involved.
11. Mouth suction is never used to fill a pipette.
12. Never force glass tubing through cork or rubber stoppers without proper lubrication.
13. Never direct the open end of test tube toward yourself or anyone else.
14. Never pour water into concentrated acid.
15. Learn the proper procedure for igniting and operating a laboratory burner. Always extinguish the flame when the burner is not being used. Make sure that all flammable reagents are well removed before lighting the burner.
16. Liquid and solid waste containers must be properly used at all times.

17. Never place chemicals directly on the balance pan. Always use a proper weighing container when using a balance to weigh a chemical. Never pour chemicals directly over the balance.
18. Never return unused chemicals to their original container (unless directed to do so by the instructor).
19. Securely replace lids, caps, and stoppers after removing reagents from containers.
20. Always wipe spatulas clean before and after inserting into reagent bottles.
21. Report any accident and/or injury, however minor, to your instructor immediately.
22. Never place anything that is not directly required for the experiment on laboratory desks; other items may interfere with the experiment.
23. All personal belongings should be placed in the bookcases as you enter the laboratory.
24. Clean up any spill immediately.
25. Before leaving the laboratory, make sure your work area is clean and dry. Ensure that all gas, water, vacuum, and air valves are completely turn ed off.
26. Your instructor is available for any assistance you may need. Never hesitate to ask questions especially if there is any question concerning proper operating procedure. Be sure that you understand every instruction before proceeding.

Eye Safety

1. Know where the nearest eye wash station is located and how to operate it.
2. Eye goggles should be worn:
    a. When working with certain caustic reagents and/or solvents, or concentrated acids and bases.
    b. When performing procedures that are likely to generate droplets/aerosols of blood or other body fluid.
    c. When working with reagents under pressure.
    d. When working in close proximity to ultra-violet radiation (light).
3. Wearing contact lenses in the laboratory is discouraged and requires extra precaution if worn. Gases and vapors can be concentrated under the lenses and cause permanent eye damage. Furthermore, in the event of a chemical splash into an eye, it is often nearly impossible to remove the contact lens to irrigate the eye because of involuntary spasm of the eyelid. Persons who must wear contact lenses should inform their supervisor to determine which procedures would require wearing no-vent goggles.

Toxic and Corrosive Materials (acids and alkali)

Toxic or Poison Hazard

Corrosive Hazard

1- To avoid dangerous splatter, ALWAYS ADD ACID TO WATER!

2.Toxic materials should be labeled with special tape when used in compounded reagents and stored in separate containers. These materials should be handled carefully and kept in the hood during preparation.

3.Acids and alkali should be carried by means of special protective carriers when transported.

4.Acid and alkali spills should be covered and neutralized by using the material from the 'spill bucket'. All material, spill and compound, should be swept up and placed in a plastic bucket for proper disposal.

5. In case of spillage, wash all exposed human tissue (including eyes) generously with water and notify your supervisor for proper reporting of the incident.

Carcinogens Chemicals

1. All laboratory chemicals identified as carcinogens must be labeled CARCINOGEN.

2.When working with these substances, protective clothing and gloves should be worn.

Flammable Compounds

1. All flammable reagents should be kept in the flammable storage facilities (closet or refrigerator) at all times when not in use.

2. Any solutions compounded from these reagents should be labeled as flammable.

3. Flammable substances should be handled in areas free of ignition sources.

4. Flammable substances should never be heated using an open flame.

5. Ventilation is one of the most effective ways to prevent accumulation of explosive levels of flammable vapors. An exhaust hood should be used whenever appreciable quantities of flammables are handled.

6. Flammable compounds should be placed in proper receptacle for disposal.
7. When ether containers are opened, they are to be dated and all material remaining after six (6) months must be disposed of immediately.

Compressed Gases

1. The storage of all compressed gases shall be in containers designed, constructed, tested and maintained in accordance with the department of Transportation Specifications and Regulations.
2. In the laboratory, gas containers are to be limited to the number of containers in use at any time. Low pressure (LP) gases shall also be limited to the smallest size container.
3. Containers shall be securely strapped, chained or secured in a cylinder stand so they cannot fall.
4. Oxidizing gases should be separated from flammable gasses.

Radiation Safety

1- No eating, drinking, smoking permitted!
2- Radioactive material should be labeled as radioactive and stored in a proper container so as to prevent spillage or leakage.
3- These materials must be handled carefully. Remember: the amount of radiation exposure decreases with distance.
4- Radioactive spills should be absorbed with absorbent toweling. The area should be cleaned with soap and water and then decontaminated with a product. The area of the spill is then monitored for any residual radioactivity. If the area is not decontaminated, the above regimen is repeated and re-monitored.

5- In the case of a radioactive spill in a high traffic area, the area will be 'roped off' until proper decontamination has been achieved.

6- In the case of a major radioactive spill, all personnel in the area must be notified. The appropriate safety officer must be notified and all attempts to keep contamination at a minimum must be used.

Electrical Safety

1. The use of extension cords is prohibited.
2. All equipment must be properly grounded.
3. Never operate electrical equipment with fluid spillage in the immediate are or with wet hands.

4. Never use plugs with exposed or frayed wires.
5. If there are sparks or smoke or any unusual evens occur, shut down the instrument and notify the manager or safety officer. Electrical equipment that is not working properly should not be used.
6. If a person is shocked by electricity, shut off the current or break contact with the live wire immediately. Do not touch the victim while he is in contact with the source of current unless you are completely insulated against shock.

LAB PROCEDURES

The following are procedures that must be followed for all lab sessions. Many are safety issues as well.

1- Dress properly for lab. Wear clothing that covers as much skin as possible. Sandals are not permitted. If possible, wear older clothes on lab day. All loose clothing and long hair must be confined.

2- Read the entire experiment before coming to lab. The instructor will briefly discuss the experiment at the beginning of each lab.

3- Arrive on time for lab. If a student arrives late for lab and misses a substantial portion of the introductory discussion and safety precautions, the student may be a threat to themselves and others in the lab. The instructor may deny the student the privilege of completing the assigned experiment.

4- Work independently unless otherwise instructed.

5- Keep the benchtop uncluttered. Only those personal items pertinent to the lab work (lab manual, etc.) are to be on the benchtop at student's workstation. Book bags, coats, etc. are not to be placed on the benchtop or on the floor close to the lab benches. Place all such items in the designated areas near the entrance to the lab.

6- Keep drawers closed. Drawers and cabinets are to be kept closed except when items are being taken from or returned to these drawers.

7- Take only planned breaks. If the need arises to take a short break, you may do so at any time during the experiment with these points in mind; try to plan the break during a less critical time in the experiment (e.g. while something is cooling); make sure that your hot plate is turned off; inform a neighbor and the instructor.

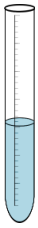
8- Do not come to lab under the influence of drugs. If, in the judgement of the instructor, a student presents a safety hazard to himself or his fellow students because the student is affected by medication, alcohol or other factors, the instructor may refuse to allow the student to continue working in the lab that day. If the situation is noted more than once, the student may be permanently removed from the course.

9- Clean up at the end of lab. At the end of all lab sessions return clean glassware to your drawer, clean your benchtop and finally wash your hands thoroughly. Be sure all electrical devices and water are turned off.

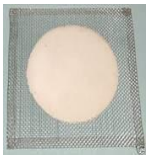
Chemistry Lab Tools

In most labs, you'll encounter the same basic apparatus. Here, you will find a picture and an explanation for how to use each piece of equipment. You will learn about:

Equipment Name	**Description of use**	**Representative Image**
Erlenmeyer flask or Conical flask	Used for titration or filtration of liquids and to prevent air contamination to sample during work.	
Round bottom flask (Florence flask)	Used for distillation or heating of liquid, allows uniform heating.	
Volumetric flask	Used for measuring liquid with high accuracy	
Filtering flask	That is used for receiving a filtering liquid.	

Beaker	Used for measuring liquid roughly volume with low accuracy	
Test Tube	used by chemists to hold, mix, or heat small quantities of solid or liquid chemicals, especially for qualitative experiments	
Graduated Cylinder	Used for measuring liquid with better accuracy than beaker	
Watch glass	Used for air drying or oven drying of liquid	
Funnel	Used for liquid transfer. Also for simple filtration	
Burette	Used in titrations to measure precisely how much liquid is used.	

Powder funnel	used for transferring aqueous solutions.	
Buchner funnel	used in filtration	
Separating funnel	Used for Liquid-Liquid extracts, designed for increase separation efficiency	
Mohr pipette	used to measure the volume of the liquid dispensed, although not as accurately as a volumetric	
Dropper	Used for transfer liquid drop by drop	
Desiccator	Used for store material and protect it from air contamination or humidity	

Pipette bulb	Used along with pipette to suck liquid	
Wire gauze	Used for spread the head of burner homogeneously	
Water Bath	Sued to incubate samples in water	
Utility Clamp	Attaches to ring stand; Supports flasks/test tubes	
Test Tube Brush	Used to clean glassware	
Spatula	It is used to take and handle small quantities of solid chemicals. It is used like a spoon or an instrument for scooping material out of a container	

Practical Experiments

In this book we will study the following experiments:

1- Acid-Base Titration

2- Redox Titration

3- pH measurement and application in acid base titration.

4- Predicting heating and cooling curves and interrelating with phase diagram.

5- Molecular weight determination from general properties of solutions.

6- Determination of solubility and evaluating solubility product constant (Ksp).

7- Determination of acid and base constants for weak acids (ka) and for weak bases (kb).

8- Determination of dissolved oxygen in water.

9- Determination of iron in cement powder.

Experiment (1) Acid-Base Titration

Theory:

In chemistry a solution is a homogeneous mixture composed of two or more substances. In such a mixture: a **solute** is dissolved in another substance, known as a **solvent**. An aqueous solution is a solution in which the solvent is water. Concentration is the measure of how of a given substance (solute) there is mixed with another substance (solvent). There are a number of different ways to quantitatively express concentration; in this work we will use molar concentration. Molar concentration (molarity) denotes the number of moles (n) of a given substance per liter (resp. dm^{-3}) of solution:

$$c = \frac{n}{V} \quad (\text{mol dm}^{-3} \text{ or M}) \quad \text{or} \quad c = \frac{m}{MV}$$

where

V is the volume of solution (in dm^3 or mL)

m is the mass of a given substance (in grams)

M is the molar mass (in g mol^{-1})

Titration is a common laboratory method of quantitative/chemical analysis that can be used to determine the unknown concentration of a known reactant (analyte). The basis of the method is a chemical reaction of a standard solution (titrant) with a solution of an analyte. The analyte (described A) is a solution of the substance whose concentration is unknown and sought in the analysis. The titrant (described T) is a solution in which the concentration of a solute is precisely known.

Because volume measurements play a key role in titration, it is also known as volumetric analysis. Usually it is the volume of the titrant required

to react with a given quantity of an analyte that is precisely determined during a titration. Using a calibrated burette (see tools above) to add the titrant, it is possible to determine the exact amount that has been consumed when the endpoint of titration is reached. The **endpoint** is the point at when the titration is complete, as determined by an indicator.

At the titration endpoint, the quantity of reactant in the titrant added during the titration is stoichiometrically equivalent to the quantity of reactant in the analyte. This is ideally the same volume as the **equivalence point** the volume of added titrant at which the number of moles of titrant (n_T) is equal to the number of moles of analyte(n_A), is in stoichiometric ratio of the given chemical reaction.

Titrations can be classified by the type of reaction. Different types of titration reaction include acid-base titrations, complexometric titrations, Redox titration and precipitation titration.

Acid-Base titrations are based on the neutralization reaction between the analyte and an acidic or basic titrant. These most commonly use a pH meter, or a conductance meter to determine the endpoint. In our experiments we will use a pH indicator to detect the endpoint of the reaction. Neutralization is a chemical reaction, also called a water forming reaction, in which an acid and a base or alkali (soluble base) react and produce a salt and water:

acid + base →salt + water

For example, the reaction between hydrochloric acid and sodium hydroxide solutions:

hydrochlorid acid + sodium hydroxide → sodium chloride + water

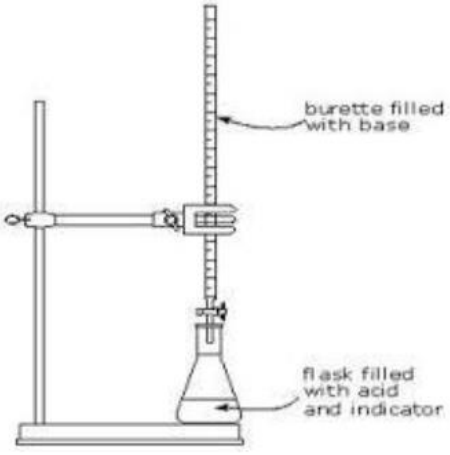
$HCl + NaOH \rightarrow NaCl + H_2O$

Before starting the titration, a suitable pH indicator must be chosen. The endpoint of the reaction, when all the products have reacted, will have a pH dependent on the relative strengths of the acids and bases. The pH of the endpoint can be roughly determined using the following rules:

- A strong acid reacts with a strong base to form aneutral (pH=7) solution.
- A strong acid reacts with a weak base to form an acidic (pH<7) solution.
- A weak acid reacts with a strong base to form a basic (pH>7) solution.

When a weak acid reacts with a weak base, the endpoint solution will be basic if the base is stronger and acidic if the acid is stronger. If both are of equal strength, then the endpoint pH will be neutral.

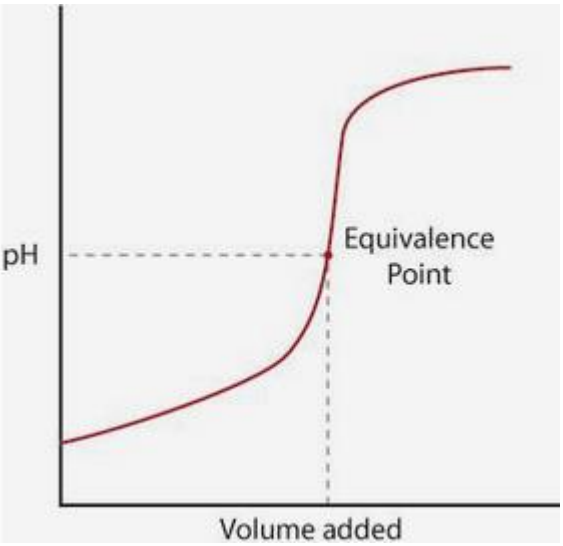
Frequently, during a titration it is also useful to monitor the progress of the titration with a graph. This graph is known as a **titration curve**. Such a curve reflects the changes in pH that occur as titrant is added from a burette to the analyte in the beaker below the burette (Figure2).



Titration Curves

1- The first curve shows a **strong acid** being titrated by a **strong base**.

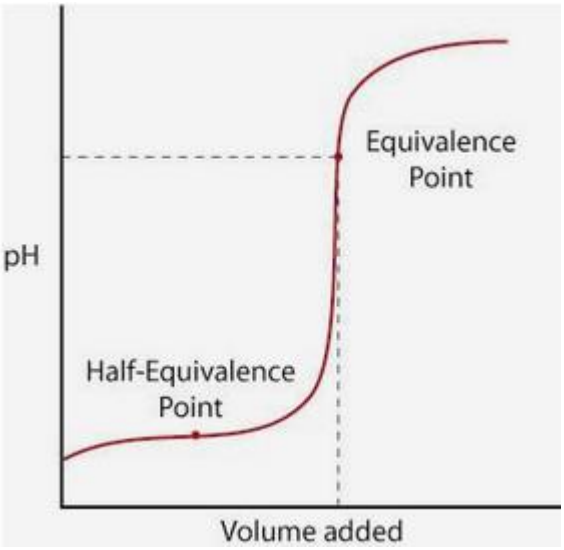
There is the initial slow rise in pH until the reaction nears the point where just enough base is added to neutralize all the initial acid. This point is called the equivalence point. For a strong acid/base reaction, this occurs at pH = 7. As the solution passes the equivalence point, the pH slows its increase where the solution approaches the pH of the titration solution.



2- The second curve shows a **weak acid** being titrated by a **strong base**

A **weak acid** only partially dissociates from its salt. The pH will rise normally at first, but as it reaches a zone where the solution seems to be buffered, the slope levels out. After this zone, the pH rises sharply through its equivalence point and levels out again like the strong acid/strong base reaction.

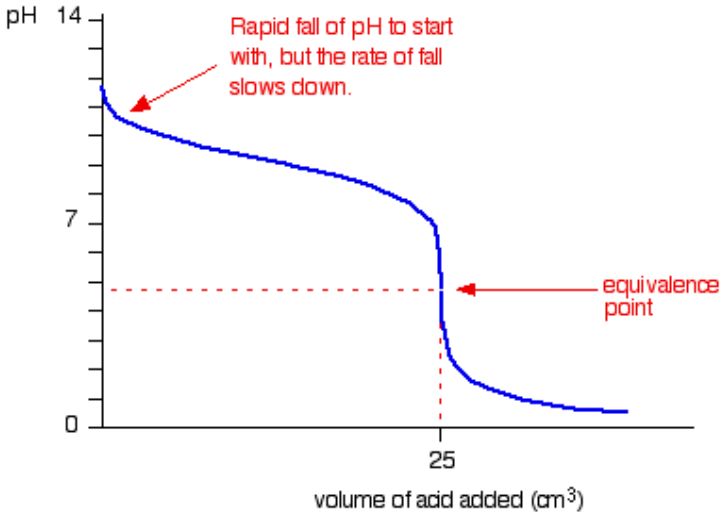
There are two main points to notice about this curve. The first is the half-equivalence point. This point occurs halfway through a buffered region where the pH barely changes for a lot of base added. The half-equivalence point is when just enough base is added for half of the acid to be converted to the conjugate base. When this happens, the concentration of H^+ ions equals the Ka value of the acid. Take this one step further, pH = pKa.



The second point is the higher equivalence point. Once the acid has been neutralized, notice the point is above pH=7. When a weak acid is neutralized, the solution that remains is basic because of the acid's conjugate base remains in solution.

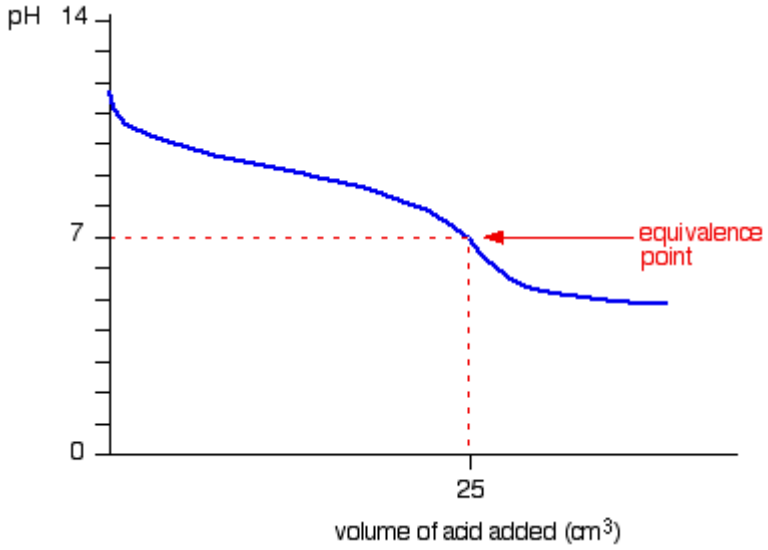
3- The third curve shows a strong acid being titrated by a weak base

In a weak base-strong acid titration, the acid and base will react to form an acidic solution. A conjugate acid will be produced during the titration, which then reacts with water to form hydronium ions. This results in a solution with a pH lower than 7. An example of this is the titration of hydrochloric acid (strong acid) into ammonia (weak base), which forms the conjugate acid ammonium and produces an acidic solution.



4- The third curve shows a weak acid being titrated by a weak base

This is the reaction of ethanoic acid vs ammonia. As you can guess, the steepest point in the curve is now harder to identify since the pH is not changing as rapidly. Of course, we can graph the derivative of the curve for more accurate determination. However, we can't simply just look at the curve to point out the equivalence point as before**.**



Another way you probably learn to perform a titration is not to track its pH but to use an indicator. We know the endpoint is defined as when the solution changes color (like colorless to pink). A problem with weak acid/weak base titration is the lack of available indicator to accurately determine its end point.

Reagent and Materials

1- burette with burette clamp
2- 10 mL graduate cylinder,
3- Erlenmeyer flask
4- Funnel
5- Volumetric flask
6- Pipette
7- Spatula
8- Glass rod
9- Washing bottle
10- Hydrochloric acid HCl (unknown concentartion)

11- 0.10 M Sodium Hydroxide (NaOH)

12- Phenolphthalein as indicator(ph.ph)

Procedure

1- THIS LABORATORY EXERCISE WILL BE DONE INDIVIDUALLY. Solutions, except the unknown, will be shared at a table. All calculations should be done in class and the Report page will be handed in at the end of the period. You will be graded on lab procedure, neatness of the report and on the accuracy of your results.

2 .Rinse out the burette with water and make sure the liquid flows through easily. Rinse the buret with a little of the 0.10 M NaOH as demonstrated.

3 .Fill the burette with the base. Let a little run out into a waste beaker. Read the volume (the bottom of the meniscus). Record .

4 .Add between 5 – 10 mL of 0.10 M HCl to a graduate cylinder and record the exact volume to the nearest 0.1 mL. Pour the HCl into an Erlenmeyer flask and add 1 drop of phenolphthalein .

5 .Add base slowly to the flask with shaking, as demonstrated, until the indicator just turns A LIGHT PINK COLOR AND THE COLOR REMAINS. This is the end-point or the neutralization point. Read and record the final volume of the buret .

6 .Repeat steps 4 and 5 two more times, for a total of three trials. It is not necessary to fill the burette each time. Rinse the flask with water after each trial. It is not necessary to dry the flask.

7 .Calculate the molarity of the acid for each trial. If the molarities is close you have mastered the technique.

Results and Calculations

No.	Volume of NaOH added (mL)	Average
1	V_1 =	
2	V_2 =	
3	V_3 =	

V_{NaOH}(Average) = = = ml

$$(N.V)_{NaOH} = (N.V)_{HCl}$$

$$N_{HCl} = \frac{N_{NaOH} \times V_{NaOH}}{V_{HCl}} = \frac{\times}{10} = \text{N}$$

Strength of HCl solution = $N_{HCl} \times$ Eq.Wt $_{HCl}$

=

= g/L

---------- *The End* ----------

Experiment (2) Reduction and Oxidation titration "Redox Titration"

In redox systems, the titration method can be adopted to determine the strength of a reductant/oxidant using a redox sensitive indicator. Redox titrations involving potassium permanganate are called permanganometric titrations. In these reactions, MnO_4^- ions acts as the self indicator.

Determination of Strength of $KMnO_4$ solution"

The strength of $KMnO_4$ solution can measured by

- Oxalic acid
- Ferrous ammonium sulphate (Mohr's salt)

Some Important Terms in Titration

1. Standard solution

A solution whose concentration is known, is called a standard solution. The substance used to prepare a standard solution is called the primary standard. Oxalic acid and sodium carbonate are some examples.

2. Concentration of a solution

Concentration of a solution is defined as the amount of a solute present in a definite volume of the solvent. Concentration of a solution can be expressed in different ways.

- **Normality**: Normality of a solution is defined as the number of gram equivalent of solute per litre of the solution. It is denoted by 'N'.

$$\text{Normality} = \frac{\text{Number of gram equivalance of solute}}{\text{Volume of the solution (in liters)}}$$

- **Molarity**: Molarity of a solution is defined as the number of gram moles of the solute per litre of the solution. It is denoted by 'M'.

$$\text{Molarity} = \frac{\text{Number of gram moles of solute}}{\text{Volume of the solution (in liters)}}$$

- ✓ In this titration, potassium permanganate is the oxidizing agent and Mohr's salt is the reducing agent.
- ✓ Mohr's salt is a double salt of ferrous sulphate and ammonium sulphate and its composition is $FeSO_4.(NH_4)_2SO_4.6H_2O$. It is a primary standard.
- ✓ Ferrous ions of Mohr's salt undergo hydrolysis in aqueous solution. To prevent the hydrolysis, Conc. H_2SO_4 needs to be added to the Mohr's salt crystals during the preparation of its standard solution.
- ✓ In this titration, the MnO_4^- ion is reduced to Mn^{2+} in the presence of acid and Fe^{2+} ions of Mohr's salt is oxidized to Fe^{3+}
- ✓ The chemical reaction that occurs in this titration can be represented by the following chemical equations.

Molecular equation

$2KMnO_4 + 3H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 3H_20 + 5[O]$

$2FeSO_4.(NH_4)_2SO_4.6H_2O + H_2SO_4 + [O] \rightarrow Fe_2(SO_4)_3 + 2(NH_4)_2SO_4 + 13H_2O] \times 5$

Overall reaction: $2KMnO_4 + 8H_2SO_4 + 10FeSO_4.(NH_4)_2SO_4.6H_2O \rightarrow K_2SO_4 + 2MnSO_4 + 5Fe_2(SO_4)_3 + 10(NH_4)_2SO_4 + 48H_2O$

Ionic equation

$$MnO_4^- + 8H^+ + 5e^- \rightarrow Mn^{2+} + 4H_2O$$

$$Fe^{2+} \rightarrow Fe^{3+} + e^-] \times 5$$

Overall reaction: $MnO_4^- + 8H^+ + 5Fe^{2+} \rightarrow 5Fe^{3+} + Mn^{2+} + 4H_2O$

$$\frac{\text{Molarity of } KMnO_4 \times \text{Volume of } KMnO_4}{\text{Molarity of Mohr's salt} \times \text{Volume of Mohr's salt}} = \frac{\text{No. of moles of } KMnO_4}{\text{No. of moles of Mohr's salt}} = \frac{2}{10}$$

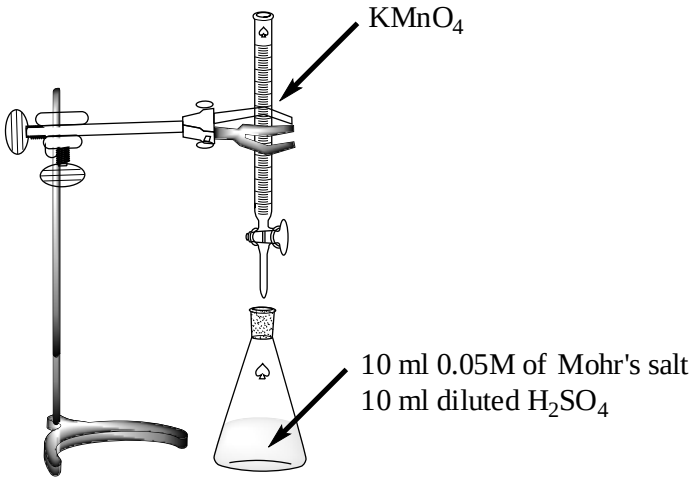
$$\text{Therefore, Molarity of } KMnO_4 = \frac{\text{Molarity of Mohr's salt} \times \text{Volume of Mohr's salt} \times 2}{\text{Volume of } KMnO_4 \times 10}$$

Practical View

1. Tools, chemicals and equipment

Chemicals	Tools and Glasses
$KMnO_4$ Solution	Burette 25ml or 50ml
Mohr's solution	Burette Stand
Diluted H_2SO_4	Conical Flask 250 ml
Distilled water	Beaker 250 ml
	Funnel
	Pipette 10 ml

2. Titration $KMnO_4$ against Mohr's salt as the following figure



Color will change from colorless to Pink color

3. Record your observation at which the color change from colorless to pink color ($KMnO_4$ color).

Results and Calculations

Trial	V_i	V_f	V
1			
2			
3			
volume			

Calculate the concentration of $KMnO_4$ as following:

$$\frac{\text{volume of } KMnO_4 * \text{ Molarity of } KMnO_4}{\text{volume of Mohr } * \text{ Molarity of Mohr}} = \frac{\text{moles of } KMnO_4}{\text{moles of Mohr}} = \frac{2}{10}$$

Molarity of $KMnO_4$ = ... M

Strength of $KMnO_4$ = Molarity of $KMnO_4$ * Molar mass of $KMnO_4$

Strength of $KMnO_4$ = Molarity of $KMnO_4$ * 158 = g/l

So Strength of $KMnO_4$ = g/l

---------- *The End* ---------

Experiment (3) pH Measurement and Application in Acid-Base Titration

This experiment can called **Potentiometric Titration.**

Objective:

In this experiment, you will use a pH meter to follow the course of acid-base titrations. From the resulting titration curves, you will determine the concentrations of the acidic solutions as well as the acid-ionization constant of a weak acid.

Introduction:

You have performed acid-base titrations in the past to determine the concentration of an acidic or basic solution using a colored indicator. However, there are times when an appropriate indicator does not exist, or where the color of the solution would obscure any color change associated with the endpoint. In such cases, a pH meter can be used to monitor the acidity of the solution throughout the titration. Recall the definition of pH:

$$pH = - \log [H_3O^+]$$

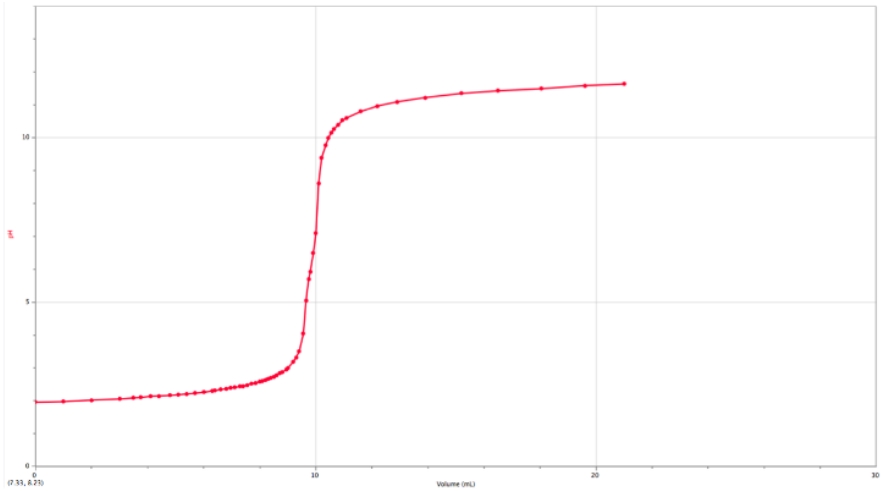
A pH meter consists of two electrodes: a glass electrode, which is sensitive to the concentration of hydronium ions in solution, and a reference electrode. The reference electrode is often a **calomelelectrode**, which supplies a constant potential ($E° = +0.24$ V

versus the standard hydrogen electrode) as determined by the half-reaction $Hg_2Cl_2 + 2\,e^- \rightleftharpoons 2\,Hg + 2\,Cl^-$

Calomel is the trivial name for the compound Hg_2Cl_2. When both the reference and glass electrodes are contained in a single unit, it is referred to as a combination electrode.

The potential of the glass electrode is proportional to the logarithm of the ratio of $[H_3O^+]$ inside and outside the electrode. The pH meter measures the total potential across the two electrodes and displays this measurement on a scale calibrated in pH units. The pH meter is an accurate and easy-to-use device for determining the pH of a solution. You will be using a pH electrode attached to computer as a pH meter in this experiment.

The following figure shows a plot of pH versus volume of base added for the titration of a strong acid with a strong base. There is very little change in pH when the base is initially added. Below the equivalence point, the pH is a function of the amount of excess acid present. Above the equivalence point, the pH is a function of the amount of excess base present. The equivalence point for the titration of a strong acid with a strong base occurs when $[OH^-]$ exactly equals $[H_3O^+]$ in the solution; pH = 7.0.



Reagent and Materials

1- pH meter
2- burette with burette clamp
3- 10 mL graduate cylinder,
4- Erlenmeyer flask
5- Funnel
6- Volumetric flask
7- Pipette
8- Spatula
9- Glass rod
10- Washing bottle
11- Hydrochloric acid HCl (unknown concentartion)
12- 0.10 M Sodium Hydroxide (NaOH)
13- Phenolphthalein as indicator(ph.ph)

Procedure

Part I. Calibrating the pH Electrode

1- To calibrate the pH electrode, you will need pH 4 and pH 7 buffer solutions.
2- Remove the pH electrode from the bottle in which it is soaking by unscrewing the cap through which the electrode is inserted. Rinse off the pH electrode with a stream of water from a wash bottle, shake off the drops and place it in the pH 7 buffer.
3- Pull down the Experiment menu and choose Calibrate followed by LabPro: 1 CH1:pH. In the box that appears, click on Calibrate Now.

Part II. Titration of a Strong Acid

1- Into a clean and dry 150 mL beaker, and with a carefully rinsed volumetric pipet, dispense a 10.00 mL aliquot of the unknown HCl solution.

2- Add exactly 75.0 mL of distilled water and 3 drops of phenolphthalein solution.

3- Fill a clean and carefully rinsed buret with the standardized 0.5 M NaOH solution (record the exact molarity from the label).

4- Record the initial buret reading in your notebook.

5- Remove the pH electrode from the buffer solution. Thoroughly rinse the electrode with distilled water, shake off the drops of water and place it in the acid solution such that the tip is immersed.

6- Stir the acid solution with the pH electrode (be careful not to break the glass tip!).

7- Now arrange the buret over the beaker so that the NaOH can be dispensed directly into the acid solution.

8- wait some time to the reading of pH meter is stable.

9- Begin the titration by adding, with stirring, 1 to 2 mL of NaOH. Be careful not to splash any liquid out of the beaker.

10- When the pH reading is stable, stop stirring, then click on the button to record the pH of the solution.

11- Continue to add base, record the pH by clicking the button and type in the total volume of NaOH added (the new reading on the buret minus the initial volume reading).

12- Slow down as you approach the equivalence point! reduce the amount of base added to 0.1 mL increments.

13- Record in your notebook the pH reading when the pink phenolphthalein endpoint color persists for 30 seconds.

14- Add increments consisting of several drops of NaOH beyond this endpoint; then increase the increments of base to 1-2 mL until 10 mL more of NaOH has been added. Record the reading of pH meter.

15- Plot graph between the amount of NaOH that added on x-axis and the change in the pH in the y-axis.

Some practical work Hints:

a. The pH values should increase in approximately 0.2 pH unit increments.

b) Be certain the solution is stirred after each addition of titrant and that the pH is stable before clicking the button.

Results and Calculations

1- Record the reading the pH against the volume of NaOH was added.

Trial	Volume of NaOH was added	Reading of pH
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		

2- Plot the required graph

3- Calculate the concentration and the strength of the unknown sample of HCl.

$$N_{HCl} = \frac{N_{NaOH} \times V_{NaOH}}{V_{HCl}} = \frac{\quad \times \quad}{10} = \quad N$$

Strength of HCl solution = $N_{HCl} \times$ Eq.Wt $_{HCl}$

= g/l

---------- *The End* ----------

Experiment (4) Predicting heating and cooling curves and interrelating with phase diagram

Purpose:

- To understand that a phase change is a physical change.
- To practice techniques of heating materials using the Bunsen burner.
- To study the effects of heating and cooling a pure substance through a change of phase.
- To construct heating and cooling curves of a pure substance using experimental data.
- To determine the freezing and melting point temperatures of the pure substance.

Discussion:

In this laboratory investigation you will take Stearic Acid and determine its freezing point and melting point experimentally.

When the data collection is completed, your graph should reflect pictorially what happens to a pure substance as its temperature is raised and lowered over a temperature interval that includes its freezing and melting points. The graph will also show how the freezing and melting points of a pure substance are related.

Safety:

Goggles and Aprons.

Equipment:

1- Sample test tube containing Stearic Acid or ice.

2- Beaker –400 mL.

3- Thermometer

4- Ring stand and Iron ring.

5- Wire gauze.

6- Test tube clamp and test tube rack.

7- Bunsen burner

8- Stop watch/timer with second hand

Procedure:

STEP ONE: Preparation for cooling curve:

Before any data can be taken the Stearic Acid test tube or Ice tube must be prepared. Therefore, it is below its freezing point. We must first melt the sample in a hot water bath to prepare it for the cooling curve data.

1- Prepare a hot water bath using a ring stand, iron ring, wire gauze, Bunsen burner and a 400 mL beaker. Fill 400 mL beaker half –way. YOU WILL PRESERVE THIS BATH!

2- Remove rubber stopper and support sample test tube in the hot water bath using your test tube holder. Now, the sample will begin to melt and turn to a liquid.

3- While the sample is melting, place the thermometer into the test tube CAREFULLY! Look for a temperature reading of about 80°C. Continue melting until a temperature of95°C is reached.

4- Now the sample is ready for the cooling curve experiment.

STEP TWO: The Cooling Curve:

1- Remove test tube sample from the hot water bath and place in into a test tube rack. This is time = 0. Record initial temperature in appropriate place on data table.

2- Continuously stir the sample with the thermometer and make temperature reading every 30 seconds. Record your temperature readings on your data table. Continue this process until a temperature of 30°C is reached. BE CAREFULL STIRRING! At some point the thermometer will cease to move. (The substance is now frozen.)

STEP THREE: The Heating Curve:

1- Using the bath from the preparation exercise, (make sure the temperature of the bath is no higher than 90°C), place the sample test tube (thermometer in it) into the bath. This time = 0 for the heating curve. Record temperature initial in you data table.

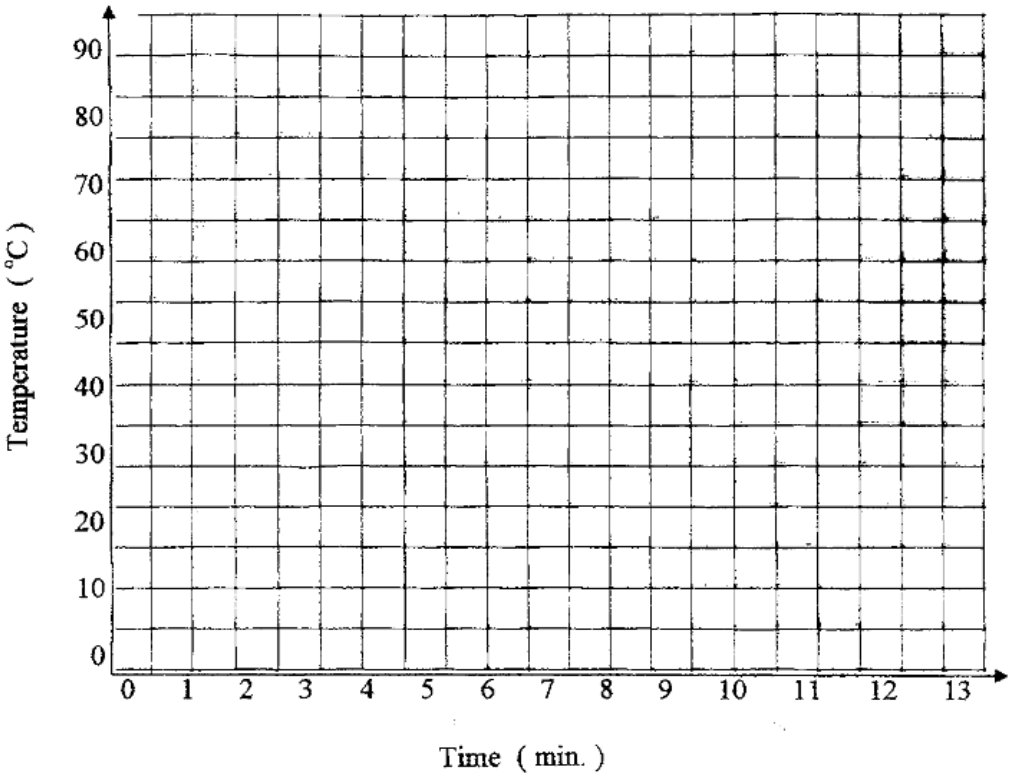
2- Continue making temperature readings every 30 seconds. Once the thermometer is free to move, make sure to stir constantly. Continue to make readings until a temperature of 90°C is achieved. (SIMMER water bath)

3. Let sample cool 2 to 3 minutes. Remove the thermometer and return the sample to its place. DO NOT DISPOSE OF THE SAMPLE!

Results and Calculations:

Time (Min)	Temperature (^{0}C)			Time (Min)	Temperature (^{0}C)	
	Cooling	Heating			Cooling	Heating
0				7		
1/2				7½		
1				8		
1 ½				8½		
2				9		
2½				9½		
3				10		
3½				10½		
4				11		
4½				11½		
5				12		
5½				12½		
6				13		
6½				13½		

Plot your data from this experiment on the set of axes in the graph below. Use a blue pencil to plot cooling data and a red pencil to plot heating data.



What phase changes are exothermic? Endothermic?

In which phaseof a substance do its particles have the greatest average kinetic energy?

---------- *The End* ----------

Experiment (5) Molecular Weight Determination from General Properties of Solutions

Introduction:

Once a solution has been formed, the solution displays properties different from those of the solvent used to prepare it. Two common examples of solvent properties that are altered are depression of the freezing point and elevation of the boiling point. The changes in these properties are related to the number of particles present in the solution and not the type of particle present. Properties that depend only on the number of particles are referred to as colligative properties. Colligative properties such as freezing point depression can be used to calculate the molecular weight of a soluble solid. To complete this calculation, the mass of solute and solvent must be known as well as the freezing points of the pure solvent and the solution. In this experiment the molecular weight of urea will be determined.

Purpose:

The purpose of this laboratory activity is to determine the molecular weight of urea using the technique of freezing point depression.

Equipment and Materials:

1- test tube or large vial.
2- foam coffee cups.

3- 250 mL and 50 mL beaker

4- Ice

5- table salt

6- thermometer or temperature probe.

7- Urea.

8- analytical balance

Safety:

- apron and goggles**.**

Procedure:

Part I: Freezing Point of Pure Water

1- Obtain a clean, dry test tube or vial. Determine the mass of the test tube or vial using the 50 mL beaker as a support.

2- Place about 10 mL of distilled water in the test tube or vial, and reweigh.

3- Determine the mass of the water used. Record the mass of the water in the data table**.**

4- Prepare an ice bath in a foam cup with ice and table salt. Place the cup in the 250 mL beaker to give it more stability. The ice bath should be deep enough so that it is above the level of the water in the test tube or vial but well below the top. Take care not to let any of the salt or ice get into the sample of distilled water**.**

5- Place a thermometer or temperature probe in the distilled water. Take time-temperature data every half-minute until ice has formed in the test tube or vial. It is not necessary to freeze the entire sample. Record the temperature at which the sample froze**.**

6- Do not discard the sample of the distilled water, because the sample will be used in Part II**.**

Part II: Molecular Weight of the Unknown

7- Remove the test tube or vial containing the distilled water from the ice bath. Allow the ice to melt. This step can be speeded up by placing the test tube or vial in a beaker of tap water**.**

8- Weigh out approximately 1 gram of urea. Record the mass of the sample in the data table. Add the urea to the distilled water, and stir until it is all dissolved. Return the test tube or vial to the ice bath. Insert the thermometer or temperature probe**.**

9- Take time-temperature data as in Part I. Again, the sample does not have to be frozen solid in order to determine the freezing point. Record the freezing point in the data table**.**

10- Repeat the procedure (both Parts I and II)

Results and Calculations:

Trial 1	Part I
Time	Temperature

Trial 1	Part I
Time	Temperature

Items	Trial 1	Trial 2
Mass of water		
T_f water		
Mass of urea		
T_f solution		

Calculations**:**

1 **.**Using the change in freezing point, the kilograms of water used, and the freezing point constant for water, calculate the number of moles of urea used in each trial**.**

$$\Delta T_f = K_f \times \frac{\text{moles solute}}{\text{kg solvent}}$$

2 **.**Using the mass of urea and the number of moles of urea, calculate the molecular weight for each trial**.**

Number or moles = n = wt/M.wt

M.Wt = wt/n

3 **.**Calculate the average molecular weight for urea and the percent error for the trials**.**

Worksheet:

1. What differences would you expect if an ionic compound such as sodium chloride were used instead of urea?

2- Why do you not have to wait for the entire sample of water to freeze in order to determine its freezing point?

3. Why is it a good idea to measure the freezing point of the water instead of assuming that its freezing point is exactly 0o C?

4. What would have happened if a two-gram sample of urea were used in this experiment?

---------- *The End* ----------

Experiment (6) Determination of Solubility and Evaluating Solubility Product Constant (K_{sp})

Introduction:

A saturated solution of Ca(OH)2 will be made by reacting calcium metal with water, then filtering off the solids.

$$Ca(s) + H_2O \rightarrow Ca(OH)_2(s) \rightleftharpoons Ca^{2+}(aq) + 2\ OH^-(aq)$$

The concentration of dissolved hydroxide will be determined by acid-base titration with standardized HCl solution. The Ksp for $Ca(OH)_2$ will be calculated from the experimentally-determined saturation concentration of hydroxide.

The equilibrium constant for the reaction is the solubility product constant, Ksp, given by the following.

$$K_{sp} = [Ca^{2+}][OH^-]^2$$

A saturated solution of $Ca(OH)_2$ can be prepared by the reaction of calcium metal with water. Calcium is oxidized by water, yielding calcium hydroxide and hydrogen gas.

$$Ca(s) + 2\ H_2O(l) \rightarrow Ca(OH)_2(s) + H_2(g)$$

Purpose:

- Understand solubility equilibria, acid-base neutralization, and the chemistry of lime ($Ca(OH)_2$), limewater, and calcium carbonate (called limestone when occurring naturally as a mineral).
- Manipulate equations for saturation equilibria, acid-base titrations, and volumetric dilutions.

Equipment and Materials

Forceps	Beaker 250 mL	Conical Flask 250 mL
Funnel	Filter Paper	Burette
Ph.ph	$Ca(OH)_2$	HCl

Safety

Apron and Goggles.

Procedure

1- Prepare the saturated $Ca(OH)_2$ solution, use your forceps to safely add a small piece of calcium metal to 150 mL of distilled water in a beaker**.**

- If you add too much metal, a large excess of calcium hydroxide solid can form and be difficult to remove by filtration**.**
- If you add too little metal, you will not form enough calcium hydroxide to saturate the solution, which is the goal of adding the metal to the water**.**

- Calcium metal is a strong reducing agent and should not touch skin.
- You may have to score the metal to rub off the tarnish and expose a clean surface to get the reaction to start.

2- You should see the white precipitate of solid Ca(OH)2, indicating that the supernatant liquid is a saturated solution of limewater.

3- Stir the solution and allow the precipitate to settle.

4- Slowly pour the solution through a funnel lined with filter paper into an Erlenmeyer flask to collect the solid-free supernatant (saturated solution of $Ca(OH)_2$). To better separate the fine precipitate particles from the supernatant, you may use two pieces of filter paper or you may filter the solution twice. Filtration may take a long time, so make the dilutions for the rest of the experiment while you wait. Do not use vacuum filtration. Do not wash the precipitate.

5- Once the filtration is complete, immediately stopper the flask containing the supernatant. Limewater can react with carbon dioxide to yield the very insoluble calcium carbonate.

$$Ca^{2+}(aq) + 2\ OH^{-}(aq) + CO_2(g) \rightarrow CaCO_3(s) + H_2O$$

Carbon dioxide from the air acts as an acid and will neutralize some of the OH– you are measuring. Minimize your saturated solution's contact with air by stoppering your flasks once you have collected the filtrate.

6- Your filtrate must be clear. Any solid $Ca(OH)_2$ interferes with the determination of the saturation concentration of OH^- ions. As the OH^- ions are removed from the sample during titration with acid, remaining solid dissociates, putting extra OH^- into solution to satisfy equilibrium. This extra OH^- would skew determination of the saturated OH^- concentration.

7- Prepare a solution of HCl to use as your titrant**.**

- Record the exact concentration of the stock HCl solution
- Prepare 0.1 M of HCl by using $M_1V_1 = M_2V_2$.

8- Titrate the saturated $Ca(OH)_2$ solution with HCl.

9- Take 10 mL from the $Ca(OH)_2$ in conical flask and then add 2 drops of ph.ph indicator.

10- titrate the above mixture with titrant HCl and record the end point when the color of solution changed.

11- Repeat steps 9 and 10 for two another trials.

12- Clean your station well.

13- Record all data in the results and calculations section.

Results and Calculations:

No.	Volume of HCl added (mL)	Average
1	V_1 =	
2	V_2 =	
3	V_3 =	

V_{HCl}(Average) = = = ml

From the stoichiometry, you get the following.

$$[OH^-] = 2[Ca^{2+}] \text{ (after dissociation)}$$

This can be substituted into

$$K_{sp} = 1/2[OH^-]^3$$

$$(N.V)_{Ca(OH)2} = (N.V)_{HCl}$$

$$N_{Ca(OH)2} = \frac{N_{HCl} \times V_{HCl}}{V} = \frac{\times}{10} = N$$

So

Ksp =

---------- *The End* ----------

Experiment (7) Determination of Acid and Base Constants for weak acids (Ka) and for weak Bases (Kb)

Introduction:

A weak acid or a weak base only dissociates partially in an aqueous medium. For this discussion, assume that HA is a weak acid and B is a weak base. The dissociations of these and the expressions for the respective equilibrium constants are shown below:

$$HA_{(aq)} + H_2O_{(l)} \Leftrightarrow H_3O^+_{(aq)} + A^-_{(aq)} \qquad K_a = \frac{[H_3O^+][A^-]}{[HA]}$$

$$B_{(aq)} + H_2O_{(l)} \Leftrightarrow BH^+_{(aq)} + OH^-_{(aq)} \qquad K_b = \frac{[BH^+][OH^-]}{[B]}$$

A simple method to determine the Ka of HA and the K_b of B is to determine the pH of known concentrations of these solutions. Assume that 0.1 M solutions of HA and B have been provided. The pH of the solutions can be used to determine the molarity of $H3O^+$ in the respective solutions at equilibrium.

$$pH = -\log[H_3O^+]$$
$$[H_3O^+] = 10^{-pH}$$

The concentrations of $H3O^+$ and OH^- in an aqueous solution are related to the autoionization of water which can be expressed by the ionic product of water (K_W).

$$K_W = 1.0 \times 10^{-14} = [H_3O^+] \times [OH^-]$$

Materials and Equipment:

0.100 M unknown acid 0.100 M unknown base

pH meter, Two 10-mL volumetric flask,

Three 10-mL beakers

Safety

Apron and Goggles.

Procedure

1.Read the instruction manual for the operation of a pH meter.

2. Obtain a pH meter from the stockroom. The instructor will demonstrate the proper use and calibration of the pH meter.

3- Obtain approximately 10 mL of an unknown weak acid of concentration 0.100 M.

4. Measure the pH of the acid in step 3.

5- Dilute the solution of 0.100 M weak acid to obtain a solution that is 0.0100 M in 50 mL.

6- Measure the pH of the acid in step 5.

7. Dilute the solution of 0.0100 M weak acid prepared in step 5 to obtain a solution that is 0.00100 M in 50 mL.

8. Measure the pH of the acid in step 7.

9. Repeat steps 3 to 8 using 10 ml of an unknown weak base of concentration 0.100 M.

10. Use the calculated values of Ka and K_b to determine the identity of the weak acid and weak base, respectively.

Results and Calculations:

1- Dilution calculations for acid:

2- Dilution calculations for acid:

3- Measuring pH for acid and Base:

Concentration	pH	
	Acid	Base
0.1		
0.01		
0.001		

4- Calculate the ka for each concentration depend on the introduction survey.

a. 0.1 M Acid

b. 0.01 M Acid

c. 0.001 M acid

5- Calculate the ka for each concentration depend on the introduction survey.

a. 0.1 M Acid

b. 0.01 M Acid

c. 0.001 M acid

---------- ***The End*** ----------

Experiment (8) Determination of Dissolved Oxygen in Water

Introduction:

There are three methods available for measuring dissolved oxygen concentrations. Modern techniques involve either an electrochemical or **optical sensor**. The dissolved oxygen sensor is attached to a meter for spot sampling and laboratory applications or to a data logger, process monitor or transmitter for deployed measurements and process control**.**

The colorimetric method offers a basic approximation of dissolved oxygen concentrations in a sample. There are two methods designed for high-range and low-range dissolved oxygen concentrations. These methods are quick and inexpensive for basic projects but limited in scope and subject to error due to other redoxing agents that may be present in the water**.**

The traditional method is the **Winkler titration**. While this method was considered the most accurate and precise for many years, it is also subject to human error and is more difficult to execute than the other methods, particularly in the field. The Winkler method now exists in seven modified versions which are still used today.

Winkler Method

The dissolved oxygen concentration of seawater is defined as the number of milliliters of dioxygen gas (O_2) per liter of seawater ($mL\ L^{-1}$).

The chemical determination of oxygen concentrations in seawater is based on the method first proposed by Winkler (1888) and modified by Strickland and Parsons (1968). Oxygen in the water sample oxidizes iodide ion (I^-) to iodine (I_2) quantitatively. The amount of iodine generated is then determined by titration with a standard thiosulfate ($S_2O_3^{-2}$) solution. The endpoint is determined by using starch as a visual indicator. The amount of oxygen can then be computed from the titer: one mole of O_2 reacts with four moles of thiosulfate.

At the time of sampling, dissolved oxygen is fixed by the addition of Mn(II) under basic conditions, resulting in a brown precipitate, manganic hydroxide ($MnO(OH)_2$). Prior to analysis, the sample is acidified to pH 1.0-2.5. This causes the precipitated hydroxides to dissolve, liberating Mn(III) ions. Mn(III) ions oxidize previously added iodide ions to iodine. Iodine forms a complex (I_3^-) with surplus iodide ions. Iodine and the complex exist in equilibrium; thus, I_3^- serves as a reservoir of I_2. The iodine is then titrated with thiosulfate; iodine is reduced to iodide and the thiosulfate is oxidized to tetrathionate. The stoichiometric equations for the reactions described above are:

$$Mn^{+2} + 2OH^- \rightarrow Mn(OH)_2$$

$$2Mn(OH)_2 + \frac{1}{2}O_2 + H_2O \rightarrow 2MnO(OH)_2$$ oxidation of Mn(II) to Mn(III)

$$2Mn(OH)_3 + 2I^- + 6H^+ \rightarrow 2Mn^{+2} + I_2 + 6H_2O$$ oxidation of I^- to I_2

$$I_2 + I^- \leftrightarrow I_3^-$$

$$I_3^- + 2S_2O_3^{-2} \rightarrow 3I^- + S_4O_6^{-2}$$ oxidation of $S_2O_3^{-2}$ to $S_4O_6^{-2}$; reduction of I_3^- to I^-

The thiosulfate solution is not stable and therefore must be standardized with a primary standard, typically potassium iodate (KIO_3). Standardization is based on the co-proportionation reaction of iodide with iodate, thereby forming iodine. As described above, the iodine binds with excess iodide, and the complex is titrated with thiosulfate. One mole of iodate produces three moles iodine, which are consumed by six moles of thiosulfate.

$$IO_3^- + 8I^- + 6H^+ \rightarrow 3I_3^- + 3H_2O$$

$$I_3^- + 2S_2O_3^{2-} \rightarrow 3I^- + S_4O_6^{2-}$$

Sampling apparatus

a. Sample flasks: Glass stoppered dissolved oxygen bottles (115 mL nominal capacity – also called “BOD bottles”). clean and dry bottle per sample, blanks and standard.

b. Volumetric dispensers (or manual volumetric pipettes (disposable, glass)).

c. Four dispensers capable of accurately dispensing 1 mL aliquots. These should be labeled “Reagent #1”, “Reagent #2” and “Reagent #3”, and “KIO_3 Blank”.

i. One dispenser capable of accurately dispensing a 10 mL aliquot. This should be labeled "KIO_3 Standard".
ii. One dispenser capable of accurately dispensing a 50 mL aliquot. This should be labeled "Sample".
iii. Tygon tubing: long enough to reach from spigot to the bottom of the sample bottle.

Titration apparatus:

a. Titration box: a three-sided box containing the titration apparatus. The inside walls should be covered with white lab paper to aid in end point detection.
b. Magnetic stirrer and stir bars.
c. 10 mL reservoir-fill buret for thiosulfate titrations.
d. Clean, dry 125 mL Erlenmeyer flasks (one per titration – 125mL beakers can also be used).
e. Glass eye dropper bottle for starch indicator.

Reagents:

a. Reagent #1: Manganese (II) chloride (3M: reagent grade): Dissolve 600 g of $MnCl_2*4H_2O$ in 600 mL distilled water. After complete dissolution, make the solution up to a final volume of 1 liter with distilled water and then filtered into an amber plastic bottle for storage.
b. Reagent #2: Sodium iodide (4M: reagent grade) and sodium hydroxide (8M: reagent grade): Dissolve 600g NaI in 600 ml of

distilled water. If the color of solution becomes yellowish brown, discard and repeat preparation with fresh reagent. While cooling the mixture, add 320g NaOH to the solution, and make up the volume to 1 liter with distilled water. The solution is then filtered and stored in an amber glass bottle.

c. Reagent #3: Sulfuric Acid (50% v/v): Slowly add 500 mL of reagent grade concentrated H_2SO_4 to 500 mL distilled water. Cool the mixture during addition of acid.

d. Starch indicator solution (manual titration only): Place 1.0g of soluble starch in a 100mL beaker and add a little distilled water to make a thick paste. Pour this paste into 1000 mL of boiling distilled water and stir for 1 minute. The indicator should be stored in a refrigerator.

e. Sodium Thiosulfate Stock solution (0.18 M: reagent grade): Dissolve 45 g $Na_2S_2O_3*5H_2O$ and 2.5g sodium borate, $Na_2B_4O_7$ (reagent grade) for a preservative, in 1 liter of distilled water. This solution is stored in a refrigerator and used to make the working thiosulfate solution. Some variation on the method call for a 0.1N (Normal) solution, which can be purchased ready-made.

f. Sodium Thiosulfate working solution (0.018 M: reagent grade): Bring 100 mL of the sodium thiosulfate stock solution to 1000 mL with distilled water in a 1 liter volumetric flask. This solution is stored in a refrigerator and used for titrations. If a ready-made

0.1 N solution was used for the stock, a working solution of 0.01 N will be fine.

g. Potassium Iodate Standard (0.00167 M = 0.01 N: analytical grade): Dry the reagent in a desiccator under vacuum. Weigh out exactly 0.3567 g KIO_3 and make up to 1.0 liter with distilled water. It is important to note the temperature of the solution so that a precise molarity can be calculated.

Sampling:

a. Collection of water, from the Niskin bottle or bucket sample, must be done soon after the sample has been collected, preferably before any other samples have been drawn. This is necessary to minimize exchange of oxygen, which typically results in contamination by atmospheric oxygen.

b. Before each water sample is collected, open the spigot on the Niskin bottle without opening the bleeder valve. If water flows out of the spigot then air must be entering the sampling bottle through a leak and the seawater within the bottle has probably been contaminated with water from shallower depths. Record this onto the data sheet.

c. If no water flows out of the spigot, attach a rubber tube to the spigot and stick the tube all the way into the sample bottle. With the spigot open, slowly open the bleeder valve to allow water to stream out of the spigot. If the sample is being collected from a bucket, carefully immerse the BOD bottle into the water so that water gently

fills the bottle without creating bubbles. Rinse the sample bottle twice with the sample water.

d. After rinsing, fill the bottle slowly - always trying to minimize the amount of air bubbles introduced into the water. Adjust the bleeder valve to control the flow. Allow the water to overflow the neck of the bottle. Place the stopper into the bottle. Record all sample information on data sheet, and make sure BOD bottle is labeled accordingly.

e. Immediately after obtaining the water sample, the following reagents are introduced into the filled BOD bottles by submerging the tip of a pipette or automatic dispenser well into the sample: 1 mL of manganous chloride (Reagent #1), followed by 1 mL of sodium iodide-sodium hydroxide solution (Reagent #2).

Titration Procedures:

a. First, the precise concentration of thiosulfate in the titrating solution must be determined. Next, a "blank" is analyzed. Impurities in the reagents may participate in the oxidation-reduction reactions involved in the dissolved oxygen analysis and thus must be accounted for. Once the standard titer and blank have been determined, the samples can be titrated with thiosulfate delivered via the buret; the endpoint is determined visually using the starch indicator solution. Below, the procedures for standardization, blank determination, and sample analysis are described. As mentioned above, the sodium thiosulfate solution will be standardized for you.

You will make your own blank and sample determinations. If time allows, you may try performing the standardization yourself.

b. Standardization: Label one clean, empty BOD bottle "KIO_3 Standard"; add approximately 15 mL of Milli-Q water and a stir bar.

i) Carefully add 10 mL of standard potassium iodate (0.00167 M = 0.01N) from an "A" grade pipette or equivalent. Swirl to mix. Immediately add 1 mL of the 50% sulfuric acid solution (Reagent #3). Rinse down sides of flask, swirling to mix, thus ensuring an acidic solution before the addition of reagents.

ii) Add 1 mL of sodium iodide-sodium hydroxide reagent, swirl, then add 1 mL of manganese chloride reagent. Mix thoroughly after each addition. Once solution has been mixed, fill to the neck with deionized water.

iii) From the "Standard" bottle, fill the 50 mL volumetric pipet with KIO3solution.

iv) Empty the first 50 mL of solution into a waste container as a rinse.

v) Re-fill the 50 mL pipet with KIO_3 solution and empty into a clean 125-mL Erlenmeyer flask. Add a magnetic stir bar.

vi) Check the 10 mL buret to ensure that it is full of thiosulfate working solution (0.01N). Place the erlenmeyer flask under the buret and turn on the magnetic stirrer. Keep the speed of the stir bar moderate - do not create a vortex in the solution.

vii) Slowly add thiosulfate to the solution until the solution turns a pale yellow color. Stop titrating.

viii) Add three drops starch solution to the flask. Continue to titrate by adding thiosulfate drop by drop just until the solution becomes colorless.

ix) Record the volume of thiosulfate added. Repeat steps 6 through 9 until you have three readings within 0.05 mL of each other.

Blank determination:

i) Place approximately 15 mL Milli-Q water in a BOD bottle with a stir bar. Add 1 mL of the potassium iodate standard, mix thoroughly, then add 1 mL 50% sulfuric acid, again mixing the solution thoroughly.

ii) Before beginning the titration, add the following reagents: 1 mL sodium iodide-sodium hydroxide reagent (#1), rinse, mix, then add 1 mL manganese chloride reagent (#2). Fill the BOD bottle to just below the neck with Milli-Q water. Titrate to the endpoint as described for the sample analysis procedure (below).

iii) Pipet 1 mL of the standard solution into the same flask and again titrate to the end point.

iv) The difference between the first and second titration is the reagent blank. Either positive or negative blanks are possible.

Sample analysis:

i) Identify the sample you are working on by the number on the bottle and the corresponding data sheet; copy this information into your notebook.

ii) Immediately prior to analysis, add 1 ml 50% H2SO4 (Reagent #3) below the water line in the sample bottle.

iii) Cover and invert the sample bottle several times to mix the solution. The precipitate should dissolve completely and the solution should turn a deep yellow color. If some precipitate remains add a few more drops of Reagent #3 (this reagent may be added in excess of 1 ml).

iv) DO NOT ALLOW THE SAMPLE TO SIT FOR ANY LENGTH OF TIME AFTER REAGENT #3 HAS BEEN ADDED - THE SAMPLE MUST BE RUN IMMEDIATELY.

v) Fill the 50-mL volumetric pipet with sample solution and empty into the waste container (this is your pipet rinse).

vi) Re-fill the 50-mL pipet with sample and empty into a clean 125-ml Erlenmeyer flask (or beaker) and add a magnetic stir bar.

vii) Check the 10 mL buret to ensure that it is full of thiosulfate solution. Place the erlenmeyer flask (or beaker) under the buret and turn on the magnetic stirrer. Keep the speed of the stir bar moderate - do not create a vortex in the solution.

viii) Slowly add thiosulfate to the sample until the solution turns a pale yellow color. Stop titrating.

ix) Add three drops starch solution to the flask. Continue to titrate by adding thiosulfate drop by drop just until the solution turns clear and colorless.

x) Record the volume of thiosulfate added. Repeat steps vi - ix until there are three readings (of thiosulfate added) within 0.05 mL of each other.

Calculation and expression of results:

The calculation of oxygen concentration (mL O_2 L^{-1}) from this analysis follows in principle the procedure outlined by Carpenter (1965).

$$O_2\left(mL/L\right) = \frac{\left((R - R_{blank}) * V_{IO_3} * N_{IO_3} * E\right)}{\left(R_{std} - R_{blank}\right) * \left(V_{bottle} - V_{rgts}\right)} - DO_{rgts}$$

where:

R = Volume of thiosulfate used to titrate the sample (mL)
R_{std} = Volume of thiosulfate used to titrate the KIO_3 standard (mL)
R_{blank} = Volume of thiosulfate used to titrate the blank as measured above (mL)
N_{IO3} = Normality of standard KIO_3 (equiv/L) – *use 0.01 N*
V_{IO3} = Volume of KIO_3 standard (mL) – *use 10 mL*
E = 5598 mL O_2 / equiv
V_{bottle} = Volume of sample bottle (mL) – *use 250 mL*
DO_{rgts} = Oxygen added in reagents – *use 0.0017 mL O_2 /L*
V_{rgts} = Volume of reagents – *use 2 mL*

---------- ***The End*** ---------

Student Name:

The section Group:

Year:

No	Date	The Name of Experiment	Teacher Assistance Signature
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2			
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Dr. Alaa E. Hassanien

Signature**:**

Notes: ……..

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الكيمياء الهندسية

(الجزء المعملى)

للهندسة والعلوم التطبيقية

2019

دكتور

علاء الدين السيد حسانين

كيمياء – قسم العلوم الأساسية

معهد المستقبل العالى للهندسة والتكنولوجيا