गुरु घासीदास विश्वविद्यालय, बिलासपुर, (छ.ग.)

Guru Ghasidas Vishwavidyalaya, Bilaspur

A Central University established by the Central University Act 2009 No. 25 of 2009
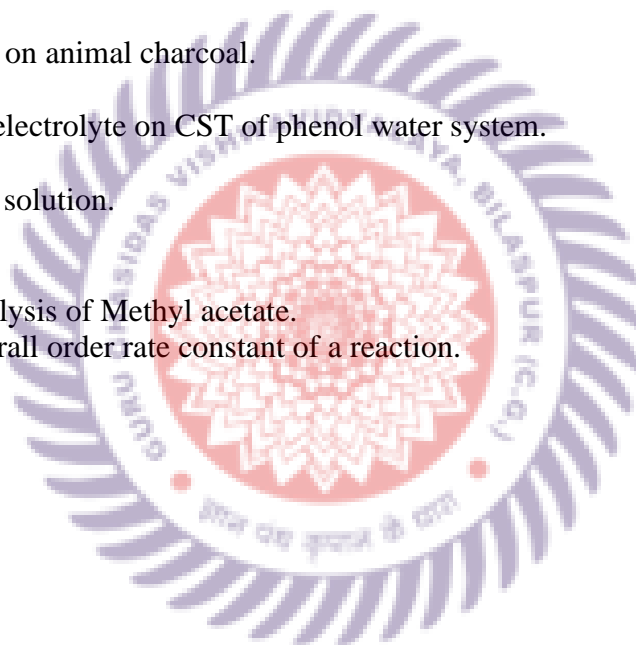
Chemistry Laboratory Manual

For

U.G. and P.G.

Department of Chemistry

Guru Ghasidas Vishwavidyalaya

Bilaspur, C.G.

A. K. Singh, P. Shriwas, GGV, Bilaspur (C.G.)

TABLE OF CONTENTS

A. K. Singh, P. Shriwas, GGV, Bilaspur (C.G.)

GENERAL CHEMISTRY LABORATORY RULES

- You are responsible for all safety rules in the laboratory.

- Keep the apparatus scrupulously clean.

- Keep the reagents in their proper places after use. Do not alter their position.

- Do not contaminate the reagents.

- Keep gas taps and water taps closed when not in use.

- Use either spirit lamp or candle to light the burner. Do not use paper torches.

- Do not throw any waste paper /litmus paper etc. into the sink. Throw them into the dust bin.

- Do not pour concentrated acids into the sink. If they are to be poured, flush them with water (liberally).

- To be a better analyst, understand the theory of the experiments you conduct.

- Record your observations as and when you proceed (and not after completion) in a note book and keep it away from reagents and sink.

- You have to wear a Lab Coat and Safety Goggles and also have a Lab Manual and Calculator while performing experiments in the laboratory. Without them you are not allowed to enter the lab section.

- Read the lab experiments and any suggested additional reading(s), before coming to lab.

- Eating, Drinking, Smoking, and Cellphones are Forbidden in the laboratory at all times. Avoid unnecessary movement and talk in the laboratory.

- Any accident involving even the most minor injury must be reported to the lab assistants.

- Do not attempt any unauthorized experiment. Perform only lab operations and activities.

A. K. Singh, P. Shriwas, GGV, Bilaspur (C.G.)

LABORATORY EQUIPMENTS

BEAKER
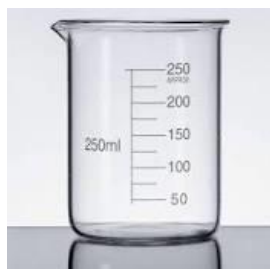
A beaker is a cylindrical glass or plastic vessel used for holding liquids. It is a multi-purpose piece of equipment used for containing a chemical reaction, measuring liquids, heating them over a Bunsen burners flame or collecting them in a titration experiment.

ERLENMAYER FLASK
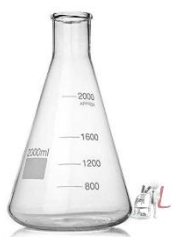
Erlenmayer flasks are conical shaped flasks, which are made of Pyrex, are safe for chemical storage, heating solutions and recrystallizations. Because of its conical shape they are best for using with equipment's with stopcocks such as separatory funnels and burettes. They are also, like beakers, for containing and transferring liquids not for making measurements.

GRADUATED CYLINDER

Graduated cylinders are used for measuring the volumes of liquids from a few milliliters to many liters. It is important to choose the graduated cylinder according to the amount of liquid to be measured for more accurate measurements. Always read meniscus point for graduated cylinders, pipettes, volumetric flasks and burettes.
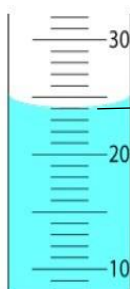
24 mL not 25 mL

What is Meniscus Point and how to read it?

Meniscus point occurs when liquid molecules adheres themselves onto the walls of the glassware. This phenomenon is known as adhesion. Always read the value right at the bottom of the meniscus

A. K. Singh, P. Shriwas, GGV, Bilaspur (C.G.)

VOLUMETRIC FLASK

Volumetric flasks are used for measuring very precise and accurate amount of a liquid and is used for such when the amount is too much for pipette or burette. They are also used for solution preparation.

PIPETTE
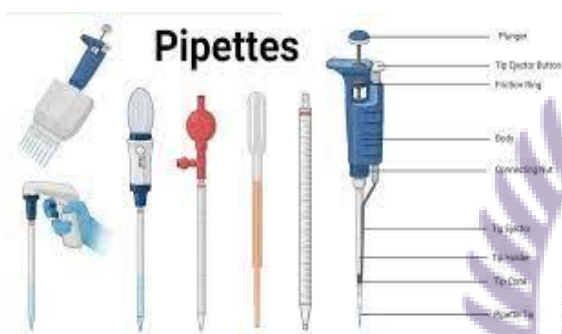
Pipette is a glass tube used for the delivery of a measured quantity of liquids. There are two kinds of pipettes:

(a) Graduated (Measuring) Pipette: has many graduated marks, much   like a graduated cylinder that can deliver moderately accurate volumes (left picture)

(b) Volumetric (Transfer) Pipette: a kind with a bulbous middle   section that has a single mark for the quantitative delivery of a single volume of liquid each time (middle and right picture)

SUCTION DEVICE

A suction device or rubber bulb is a device to place on top of pipettes for generation of vacuum to transfer known volume of liquid, usually from one container to another.

Use of a suction device with a pipette:

Before using the suction device deflate the air inside by
pressing button A. Then connect the suction device on to a
pipette. Press the button S for suction. Measure desired
amount of liquid with pipette by taking meniscus point into
account. While emptying use button E
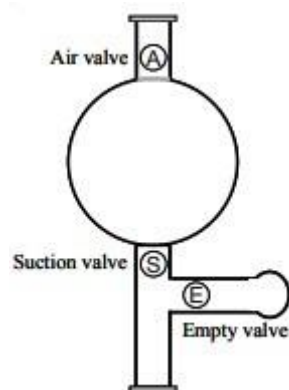
STAND

A metal rod attached to a heavy metal base. The heavy base keeps the stand
stable, and the vertical metal rod allows for easy height adjustment of the iron
ring/clamp.
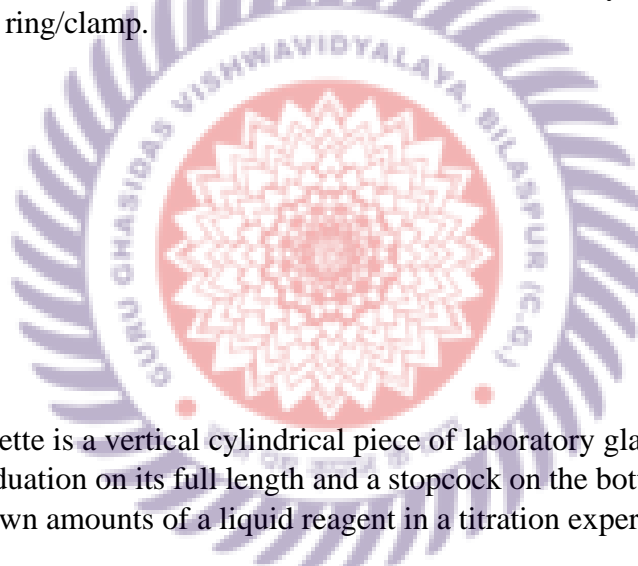
BURETTE

Burette is a vertical cylindrical piece of laboratory glassware with a volumetric
graduation on its full length and a stopcock on the bottom. It is used to dispense
known amounts of a liquid reagent in a titration experiment.

A. K. Singh, P. Shriwas, GGV, Bilaspur (C.G.)

BURETTE CLAMP

Burette clamp is used to fix the burette onto the stand.

RING SUPPORT

An iron ring/clamp that holds glassware such as a funnels and separatory funnels, and it can be attached to an iron stand.

FUNNEL

Funnels are used for pouring liquids from one container to another. In addition, with the aid of filter paper, they can be used as separation devices to separate liquids from solids. It is fixed by a ring support on a stand.

FILTER PAPER

Filter paper is used to separate solid particles from liquids. They can have different size with different pore size.

Solids that remain on filter paper can later be dried on a watch glass or in an oven.

WATCH GLASS

Watch glass is used to allow crystals to dry after they have been filtered. They can be used as an evaporating surface or to cover a beaker that can be heated to very high temperatures.

SEPARATORY FUNNEL

Separatory funnel is used in liquid-liquid extractions to separate the components of a mixture between two immiscible solvent phases of different densities. It is fixed on a stand by a ring support.

OVEN

An oven is an enclosed chamber in which heat is produced to dry chemicals or laboratory equipment's.

EVAPORATING DISH

The evaporating dishes are made of porcelain or ceramic material to heat and evaporate solutions to dryness.

ROUND-BOTTOM FLASK

Round bottom flasks are used for heating or boiling of a liquid, in distillation procedures and to carry out chemical reactions. Their two or three-necked versions are also available and usually more suitable for carrying out reactions.

TEST TUBE

Test tubes are used as containers for solids and liquids to perform quick tests for properties such as solubility, effect of heat, etc. They can also be used as centrifuge tubes when a separation of solid and liquid is necessary.

TEST TUBE RACK

The test tube racks provide places to hold the test tubes vertical so that chemicals are not spilled out.

TEST TUBE BRUSH

Test tube brush is a long and narrow equipment to clean the inside of glassware particularly test tubes.

THERMOMETER

A thermometer is a device used to measure temperatures or temperature changes.

SPATULA

Spatula or spoons are hand tools used to weigh solids in balance machine.

BALANCE Machine

A balance machine is a scale for weighing; to obtain mass of various objects.

STIRRING ROD

A stirring rod is made of solid glass and used to stir liquids in

flasks or beakers.

MAGNETIC BAR

Magnetic bars employ rotating magnetic field to mix a chemical mixture or a reaction continuously once they are placed in.
For a magnetic bar to work a stirrer must be placed below or a heater with a stirrer feature.

HEATER

A heater is a device that heats water or other solutions to a desired temperature. They usually have a stirrer feature to use with magnetic bars while carrying out a reaction.

A. K. Singh, P. Shriwas, GGV, Bilaspur (C.G.)

BUNSEN BURNER

Bunsen burner is a used for heating when no flammable material is present. The burner can be regulated by changing the air and gas mixture.

WASH BOTTLE

Wash bottles usually contains distilled water or acetone and makes for a convenient method to wash out lab glassware's.

STOPPER

Stoppers are used to close flasks and test tubes to protect it from the environments.

FUME HOODS

The fume hoods protect laboratory workers from fumes and potentially dangerous chemicals reactions by continuously vacuuming air out of the lab and providing a glass shield.

A. K. Singh, P. Shriwas, GGV, Bilaspur (C.G.)

CENTRIFUGE

Centrifuge, any device that applies a sustained centrifugal force that is, a force due to rotation. Effectively, the centrifuge substitutes a similar, stronger, force for that of gravity. Every centrifuge contains a spinning vessel; there are many configurations, depending on use.

VACUUM OVEN

A vacuum oven, or vacuum drying oven, is primarily used to expedite the drying process by employing vacuum and heat. As the pressure decreases, the boiling point of the solvent also decreases, allowing the vacuum oven to remove moisture and volatile substances at lower temperatures.

A. K. Singh, P. Shriwas, GGV, Bilaspur (C.G.)

1. **Titration between a strong acid and strong base: Titration between HCl and NaOH.**

Aim: Determine the concentration of the given strong acid (HCI) solution by titrating it with standard strong base (NaOH) conductometrically.

Requirements
Apparatus: Conductivity bridge, conductivity cell, 100 ml beaker, pipette, stand, etc.

Chemicals: HCl solution ($\approx 0.1M$), 0.5M NaOH solution.

Principle: The reaction between HCl and NaOH can be represented as:
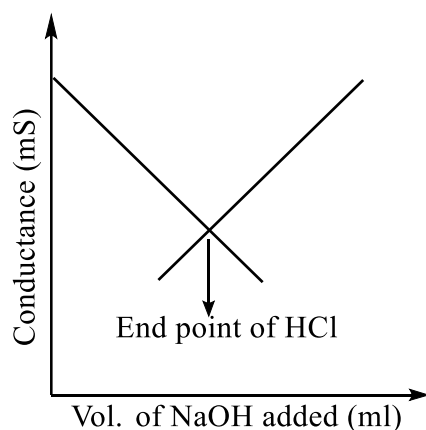
$$HCl + NaOH \longrightarrow NaCl + H_2O$$
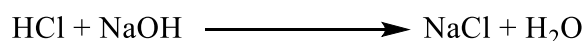
HCI undergoes reaction with sodium hydroxide resulting in the formation of sodium chloride and unionized water. At the beginning of the titration the acid solution has a high conductivity due to highly mobile hydrogen ions. When NaOH is added to HCl solution the highly mobile H+ ions are replaced by less mobile sodium ions. This will result in the decrease of conductivity rapidly. At the end point the solution will contain only sodium and chloride ions. Hence there will be minimum conductivity. After the end point the conductivity rises due to the presence of fast moving OH ions. Thus, the graph which is plotted between the conductance and the volume of sodium hydroxide added include two straight lines. The point of intersection of these two sharp lines gives the end point of the titration.

Procedure: Pipette out 40 ml of the given HCl solution into a clean 100 ml beaker and dip the conductivity cell in it. Connect the conductivity cell to a conductivity bridge and measure the initial conductance of the given HCl solution. Fill the burette with the given sodium hydroxide solution. Then add 0.5 ml of the given NaOH solution to the HCl solution present in the beaker and stir the contents well. Measure the conductance of the solution. Continue the addition of the sodium hydroxide solution in equal volumes (0.5 ml) until you get a minimum of 25-30 conductance readings. The end point of the titration can be detected by plotting a graph in between the measured conductance and the volume of sodium hydroxide that is added during the titration.

Observation Table:

S. No.	Volume of NaOH	Conductance (mS)

A. K. Singh, P. Shriwas, GGV, Bilaspur (C.G.)

Calculations:

HCI vs NaOH

$$M_1V_1 = M_2V_2$$

Where, M_1 = Molarity of the given HCI solution
V_1 = Volume of the HCI taken in the beaker = 40 ml, M_2= Molarity of the given NaOH solution = 0.5M
V_2 = Volume of the NaOH required neutralize the given acid = end point volume

$$M_1 = M_2V_2 / V_1$$
$$M_1 = 0.5 \text{ X E.P.V.} / 40$$

Results: The concentration of the given acid solution is _____ M.

Precautions:
1. Always wear appropriate eye protection (i.e., chemical splash goggles) in the laboratory.
2. Wear disposable gloves, when handling hazardous materials.
3. Wear a full-length, long-sleeved laboratory coat or chemical-resistant apron.
4. Wear shoes that adequately cover the whole foot; low-heeled shoes with non-slip soles are preferable.
5. Clean apparatus before and after use.

2. **Titration between a weak acid and strong base: Titration between CH₃COOH and NaOH.**

Aim: Determine the concentration of the given weak acid (CH_3COOH) solution by titrating it with standard strong base (NaOH) conductometrically.

Requirements:
Apparatus: Conductivity Bridge, conductivity cell, 100 ml beaker, pipette, stand, etc.

Chemicals: CH_3COOH solution (≈ 0.1M) 0.5M NaOH solution.

Principle: The reaction between acetic acid and sodium hydroxide can be represented as:

$$CH_3COOH + NaOH \longrightarrow CH_3COONa + H_2O$$
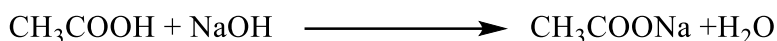
At the beginning of the titration the conductance of the solution is found to be less because of poor dissociation of the acetic acid, which is a weak acid. Thus, the number of ions produced by the dissociation of the acid is found to be less. When small amount of NaOH is added to the acetic acid solution the conductance increases due to the formation of sodium acetate which is a strong electrolyte than the acetic acid, hence dissociates rapidly producing a greater number of ions than acetic acid. At the end point of the titration both the sodium and acetate ions are present in the solution. The conductance of the solution increases when further sodium hydroxide is added to the solution at the end point. This increase is due to the presence of fast moving OH ions.

Procedure: Pipette out 40 ml of the given CH_3COOH solution into a clean 100 ml beaker and dip the conductivity cell in it. Connect the conductivity cell to a conductivity bridge and measure the initial conductance of the given CH_3COOH solution. Fill the burette with the given sodium hydroxide solution. Then add 0.5 ml of the given NaOH solution to the CH_3COOH solution present in the beaker and stir the contents well. Measure the conductance of the solution. Continue the addition of the sodium hydroxide solution in equal volumes (0.5 ml) until you get a minimum of 25-30 conductance readings. The end point of the titration can be detected by plotting a graph in between the measured conductance and the volume of sodium hydroxide that is added during the titration.

Result: The concentration of the given acetic acid solution is _____ M.

Observation Table:

S. No.	Volume of NaOH	Conductance (mS)

End point of Acetic acid

Volume of NaOH

A. K. Singh, P. Shriwas, GGV, Bilaspur (C.G.)
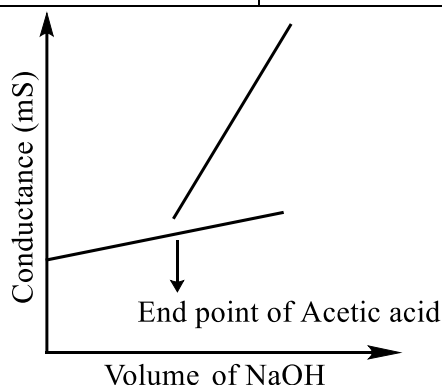
Calculations:

CH₃COOH vs NaOH

$$M_1 V_1 = M_2 V_2$$

Where, M_1 = Molarity of the given CH₃COOH solution

V_1 = Volume of the CH₃COOH taken in the beaker = 40 ml,

M_2 = Molarity of the given NaOH solution = 0.5M

V_2 = Volume of the NaOH required neutralize the given acid = end point volume

$$M_1 = M_2 V_2 / V_1$$
$$M_1 = 0.5 \text{ X E.P.V. } / 40$$

Results: The concentration of the given acid solution is _____ M.

Precautions:
1. Always wear appropriate eye protection (i.e., chemical splash goggles) in the laboratory.
2. Wear disposable gloves, when handling hazardous materials.
3. Wear a full-length, long-sleeved laboratory coat or chemical-resistant apron.
4. Wear shoes that adequately cover the whole foot; low-heeled shoes with non-slip soles are preferable.
5. Clean apparatus before and after use.

3. **Determination Of Cell Constant.**

   Aim: To determine the cell constant for a given cell at room temperature.

   Requirements
   Apparatus: Beaker, Pipette, Standard flask-100ml, Weight box.

   Chemicals: N/10 KCl Solution

   Principle: Cell constant for a cell is defined as the constant factor which stands for the ratio of the specific conductance of a solution and its measured conductance in the cell.

   Specific conductance / measured conductance = Cell constant

   Or Specific conductance = measured conductance x Cell constant.

   Since for any conductor the resistance R = p 1/a

   Taking reciprocals   1/R = 1/p xa/1

   Or 1/p = 1/R x 1/a

Therefore, specific conductance = conductance x 1/a and cell constant = 1/a

Procedure: Prepare 0.1M KCl solution by weighing accurately 0.7455gm of KCI into a clean 100ml standard flask. From this 0.1M KCl solution prepare 100ml each of 0.05M, 0.02M, 0.01M, and 0.001M KCl solutions. Take about 40ml of each solution in to a clean and dry 100ml beaker and dip the conductivity cell and make necessary connections. Measure the conductance of each solution and note down.

Note the specific conductance values of each of the solution from literature. The Cell constant is calculated by using the formula given.

Observation Table:

S. No.	Concentration	Observed Conductance	Specific Conductance	Cell Constant

Result: The cell constant was observed to be _____.

Precautions:
1. Always wear appropriate eye protection (i.e., chemical splash goggles) in the laboratory.
2. Wear disposable gloves, when handling hazardous materials.
3. Wear a full-length, long-sleeved laboratory coat or chemical-resistant apron.
4. Wear shoes that adequately cover the whole foot; low-heeled shoes with non-slip soles are preferable.
5. Clean apparatus before and after use.

4. Determination of dissociation constant of a weak acid.

Aim: To determine the dissociation constants of acetic acid and to verify the Ostwald's dilution law

Requirements
Apparatus: Conductivity meter, Conductivity cell, Pipette, Glass rod, Beaker

Chemicals: 0.1M CH_3COOH, distilled water.

Principle: An acid dissociation constant, K_a, (also known as acidity constant, or acid-ionization constant) is a quantitative measure of the strength of an acid in solution. Each acid has a different pK_a. It is the equilibrium constant for a chemical reaction known as dissociation in the context of acid-base reactions. The larger the K_a value, the more dissociation of the molecules in solution and thus the stronger the acid. Thus, a strong

acid "wants" to get rid of its hydrogen ion, much more so than a weak acid. A small amount of strong acid in water will lead to a low pH whereas the same concentration of a weak acid will not lead to such a low pH. Owing to the many orders of magnitude spanned by K_a values, a logarithmic measure of the acid dissociation constant is more commonly used in practice.

$$\text{The logarithmic constant, } pK_a = -\log K_a$$

PK, is sometimes also referred to as an acid dissociation constant. The larger the value of pKa, the smaller the extent of dissociation at any given pH, the weaker the acid.

According to Arrhenius theory of electrolyte dissociation, the molecules of an electrolyte in solution is constantly splitting up into ions and the ions are the dissociation constant (Kor constantly reuniting to form unionized molecules. Therefore, a dynamic equilibrium exists between ions and unionized molecules of the electrolyte in solution. It was pointed out by Ostwald that like chemical equilibrium, law of mass action van be applied to such systems also. The law is based on the fact that only a portion of the electrolyte is dissociated into ions at ordinary dilution and completely at infinite dilution.

Weak electrolytes are partially dissociated in solution. Hence for such electrolytes) is given by the Ostwald's dilution law as follows

$$K = \frac{C \, \alpha 2}{1 - \alpha}$$

Where C = the molar concentration, α = degree of dissociation

The value of 'α' is given as the ratio of the equivalent conductivity of the electrolyte at a particular concentration to that at infinite dilution. i.e. However, in such cases λ_∞ may be determined by the application of Kohlrausch's law of independent migration of ions and conductance] = 1000*k./c. [Where k = specific

Procedure:
        Determination of cell constant: Take 40mL of 0.1M KCl solution into a 100mL beaker. Dip the conductivity cell into the KCI solution which in turn connected to the Conductivity Bridge and note down corresponding observed conductance value. Now take 40 mL.0.01M KCI solution into the beaker and repeat the same experimental procedure as mentioned earlier. Calculate the cell constant using following formula
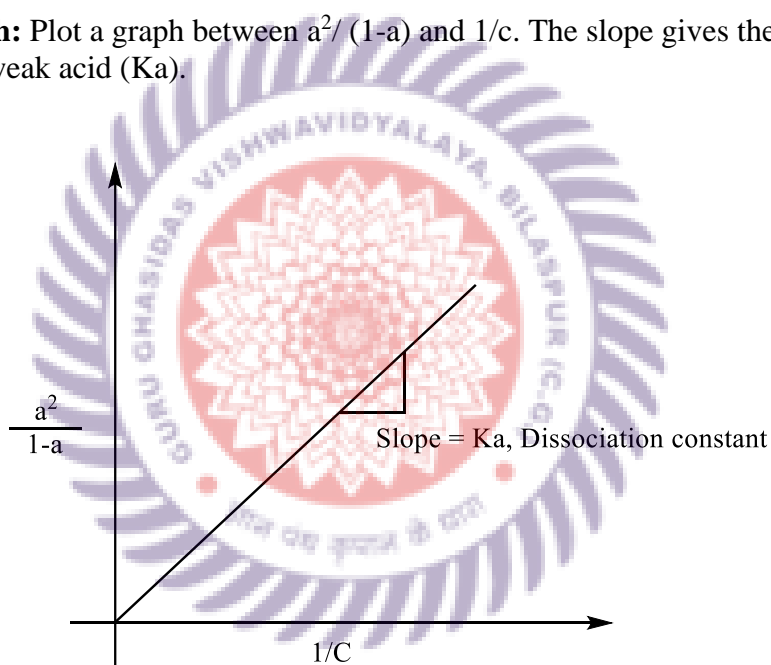
$$\text{Cell constant} = \frac{\text{Specific conductance}}{\text{Observed Conductance}}$$

A. K. Singh, P. Shriwas, GGV, Bilaspur (C.G.)

Observation Table:

S. No.	Concentration	Specific conductance (k_v) [From reference]	Observed conductance [From experiment]	Cell constant
1.	0.1 M	12.88 mS/cm		
2.	0.01M	1.413 mS/cm		

Take 40 mL 0.1M CH$_3$COOH solution in a clean beaker whose dissociation constant is to be determined. Now dip the conductivity cell whose cell constant is known which in turn connected to the Conductivity Bridge and note down corresponding conductance value. For other concentrations, remove the 20ml of this solution and add 20ml distilled water to the same. Repeat the similar procedure 2 for four more times and note down the corresponding conductance values for each dilution.

Model graph: Plot a graph between $a^2/(1-a)$ and $1/c$. The slope gives the dissociation constant of weak acid (Ka).

Slope = Ka, Dissociation constant

Model Tubular Form:

S. No.	Concentration (C)	Observed conductance (mho)	Specific conductance (k_v)	Equivalent conductance $K = \dfrac{1000\,kv}{C}$	Degree of dissociation $\alpha = \dfrac{\lambda v}{\lambda \infty}$	Dissociation constant (K_a)	$\alpha^2/(1-\alpha)$ or $a^2/(1-a)$	1/C

A. K. Singh, P. Shriwas, GGV, Bilaspur (C.G.)

1.	0.1							
2.	0.05							
3.	0.025							
4.	0.0125							
5.	0.00625							

Result: Dissociation constant of acetic acid (K_a) = _____

Precautions:
1. Always wear appropriate eye protection (i.e., chemical splash goggles) in the laboratory.
2. Wear disposable gloves, when handling hazardous materials.
3. Wear a full-length, long-sleeved laboratory coat or chemical-resistant apron.
4. Wear shoes that adequately cover the whole foot; low-heeled shoes with non-slip soles are preferable.
5. Clean apparatus before and after use.
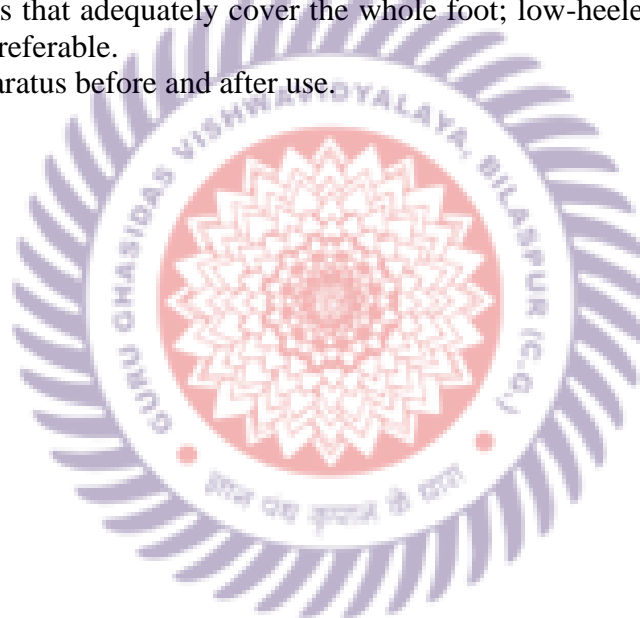
POTENTIOMETRY

1. Titration of a strong acid with a strong base - Titration of HCl against NaOH
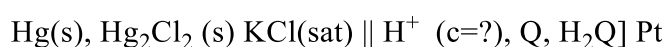
Aim: To find out the concentration of the given hydrochloric acid solution by potentiometric method.

Requirements

Apparatus: Potentiometer, beaker (100 ml), stand, pipette, reference electrode (Calomel electrode), etc.

Chemicals: HCl solution, 0.1 M NaOH Solution, Quinhydrone powder, potassium chloride, etc.

Principle: The cell which is established to determine the concentration of an acid is represented as:

$$Hg(s), Hg_2Cl_2 \text{ (s) KCl(sat)} \parallel H^+ \text{ (c=?), Q, } H_2Q] \text{ Pt}$$

The two electrodes are calomel electrode (reference electrode) and quinhydrone electrode which is a pH indicating electrode. Quinhydrone is an equimolar mixture of both quinine and hydroquinone. This electrode is developed by the addition of a pinch of quinhydrone to a solution containing H ions to which it is reversible. During the titration as concentration of H ions changes the potential of the indicator electrode, quinhydrone electrode changes. This change in potential can be determined by coupling this with a reference electrode, calomel electrode whose potential value remains constant. Thus, the cell EMF varies only with the potential of indicator electrode. Thus, end point of such titration can be obtained by plotting a graph between the measured EMF and volume of base added which causes a change in potential of indicator electrode and hence the cell EMF.

Procedure: Pipette out 10 ml of the given HCl solution into a clean beaker and dip the calomel electrode and the Platinum electrode. Now a pinch of quinhydrone is added to the HCl solution. Connect the two electrodes to a potentiometer to read the cell EMF. Fill the burette with the given NaOH solution (0. 1M). Before the addition of sodium hydroxide to the acid solution measure the EMF. Now add 1 ml of the given sodium hydroxide solution to the HCl solution present in the beaker and stir with a glass rod and measure the EMF. Continue the addition of equal volumes of base until a large change in EMF is observed. Now add 0. 2 ml of NaOH every time to get an accurate end point. Continue this addition until you get a minimum of 7-8 readings after the end point. Plot a graph between the measured EMF of the cell and volume of sodium hydroxide where in a sigmoid curve will be obtained. The inflexion of the curve i.e, where the curve changes its direction is taken as the end point of the titration. Accurate endpoint can be obtained by plotting a graph between $\Delta E/\Delta V$ against V_{avg}.

Observation Table:

S. No.	Vol. of NaOH added (ml)	EMF (mv)	ΔE	ΔV	$\Delta E / \Delta V$	V_{avg}	$pH = \dfrac{0.4595 - Ecell}{0.0591}$

<table>
<tr><td></td><td></td><td></td><td></td><td></td><td></td><td></td></tr>
<tr><td></td><td></td><td></td><td></td><td></td><td></td><td></td></tr>
</table>

Calculation:

HCl vs NaOH

$$M_1V_1 = M_2V_2$$

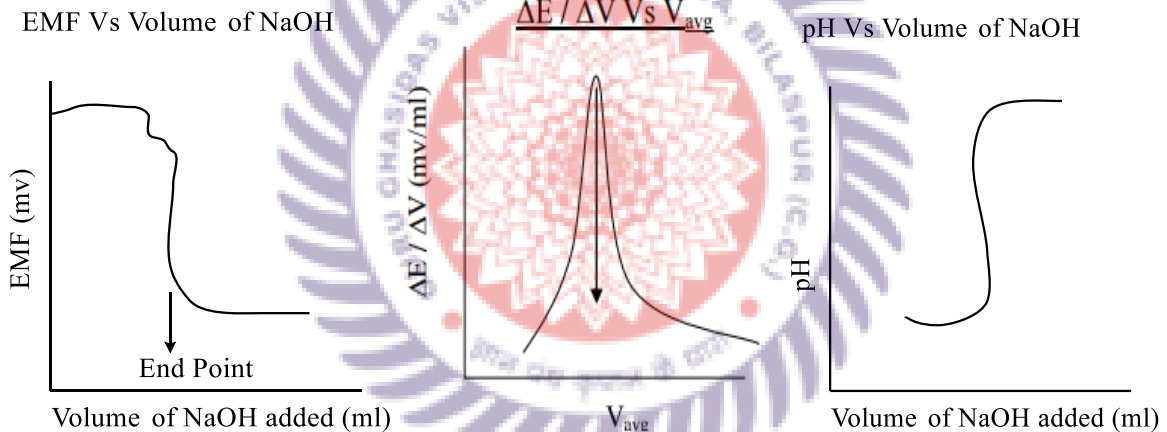Where, M_1 = Molarity of the sodium Hydroxide solution = 0.1M

V_1 = Volume of the NaOH required to neutralize the HCl solution = end point volume,

M_2 = Molarity of the given HCl solution

V_2 = Volume of the given HCl solution taken in the beaker = 10 ml

$$M_2 = M_1V_1 / V_2$$
$$M_2 = 0.1 \ X \ E.P.V. \ / \ 10$$

EMF Vs Volume of NaOH $\Delta E / \Delta V$ Vs V_{avg} pH Vs Volume of NaOH

Result: Concentration of the given HCl solution = _____ M

Precautions:
1. Always wear appropriate eye protection (i.e., chemical splash goggles) in the laboratory.
2. Wear disposable gloves, when handling hazardous materials.
3. Wear a full-length, long-sleeved laboratory coat or chemical-resistant apron.
4. Wear shoes that adequately cover the whole foot; low-heeled shoes with non-slip soles are preferable.
5. Clean apparatus before and after use.

2. **Titration of a Weak acid with a strong base - Titration of CH₃COOH against NaOH.**
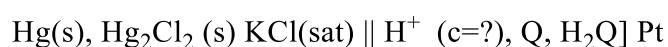
Aim: To find out the concentration of the given acetic acid solution by potentiometric method.

Requirements
Apparatus: Potentiometer, beaker (100 ml), stand, pipette, reference electrode, etc.

Chemicals: Acetic acid solution, 0. 1 M NaOH Solution, Quinhydrone powder, potassium chloride, etc.

Principle: The cell which is established to determine the concentration of an acid is represented as:

$$Hg(s), Hg_2Cl_2 \text{ (s) } KCl(sat) \parallel H^+ \text{ (c=?), Q, } H_2Q] \text{ Pt}$$

The two electrodes are calomel electrode (reference electrode) and quinhydrone electrode which is a pH indicating electrode.

Quinhydrone is an equimolar mixture of both quinine and hydroquinone. This electrode is developed by the addition of a pinch of quinhydrone to a solution containing H ions to which it is reversible. During the titration as concentration of H ions changes the potential of the indicator electrode, quinhydrone electrode changes. This change in potential can be determined by coupling this with a reference electrode, calomel electrode whose potential value remains constant. Thus, the cell EMF varies only with the potential of indicator electrode. Thus, end point of such titration can be obtained by plotting a graph between the measured EMF and volume of base added which causes a change in potential of indicator electrode and hence the cell EMF.

Procedure: Pipette out 10 ml of the given acetic acid solution into a clean beaker and dip the calomel electrode and the Platinum electrode. Now a pinch of quinhydrone is added to the acetic acid solution. Connect the two electrodes to a potentiometer to read the cell EMF. Fill the burette with the given NaOH solution (0. IM). Before the addition of sodium hydroxide to the acid solution measure the EMF. Now add 1 ml of the given sodium hydroxide solution to the acetic acid solution present in the beaker and stir with a glass rod and measure the EMF. Continue the addition of equal volumes of base until a large change in EMF is observed. Now add 0. 2 ml of NaOH every time to get an accurate end point. Continue this addition until you get a minimum of 7-8 readings after the end point. Plot a graph between the measured EMF of the cell and volume of sodium hydroxide where in a sigmoid curve will be obtained. The inflexion of the curve i.e, where the curve changes its direction is taken as the end point of the titration. Accurate endpoint can be obtained by plotting a graph between $\Delta E/\Delta V$ against V_{avg}.

Observation Table:

S. No.	Vol. of NaOH added (ml)	EMF (mv)	ΔE	ΔV	$\Delta E / \Delta V$	V_{avg}	$pH = \dfrac{0.4595 - E_{cell}}{0.0591}$

Calculation:

CH$_3$COOH vs NaOH

$$M_1 V_1 = M_2 V_2$$

Where, M_1 = Molarity of the sodium Hydroxide solution = 0.1M

V_1 = Volume of the NaOH required to neutralize the HCl solution = end point volume,

M_2 = Molarity of the given HCl solution

V_2 = Volume of the given HCl solution taken in the beaker = 10 ml

$$M_2 = M_1 V_1 / V_2$$
$$M_2 = 0.1 \text{ X E.P.V. } / 10$$

In case of a weak acid, the dissociation constant can be determined by using Henderson's equation. According to this equation.

$$pH = pKa + log\frac{[salt]}{[acid]}$$
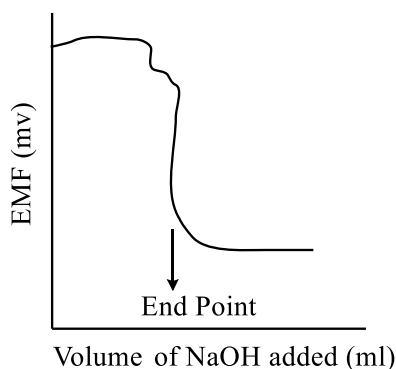
At half neutralization point,

pH = pKa

Thus, above equation reduces to

pH = pKa

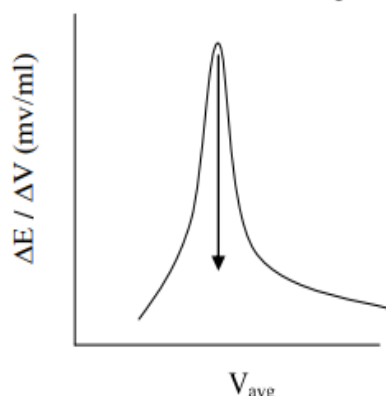and from pKa value Ka (dissociation constant) can be calculated.

pKa = -log Ka

A. K. Singh, P. Shriwas, GGV, Bilaspur (C.G.)

EMF Vs Volume of NaOH

$\Delta E / \Delta V$ Vs V_{avg}

Result: Concentration of the given acetic acid solution = _____ M.

Precautions:
1. Always wear appropriate eye protection (i.e., chemical splash goggles) in the laboratory.
2. Wear disposable gloves, when handling hazardous materials.
3. Wear a full-length, long-sleeved laboratory coat or chemical-resistant apron.
4. Wear shoes that adequately cover the whole foot; low-heeled shoes with non-slip soles are preferable.
5. Clean apparatus before and after use.

3. **Weak acid vs Strong base (Acid-Base Titration).**

Aim: To find the strength of acetic acid by titrating it against sodium hydroxide potentiometrically and also calculate the dissociation constant (K) of the acid using quinhydrone electrode.

Requirements
Apparatus: Potentiometer, calomel electrode, Pt wire, salt bridge, beakers.

Chemicals: 0.1M AcOH, 0.1M NaOH, saturated KCl, Quinhydrone etc.

Principle: Potentiometric methods of analysis are based upon measurements of the potential of electrochemical cells under conditions of zero current, where the Nernst equation governs the operation of potentiometry. A typical cell for potentiometric analysis consists of a reference electrode, an indicator electrode and a salt bridge.

To measure the potential changes, the indicator electrode is coupled with a reference electrode using a salt bridge. The cell can be depicted as follows

$$Hg\ (l),\ Hg_2Cl_2(s)\ |\ KCI\ \|\ H^+,\ Q,\ QH_2\ |\ Pt$$

Quinhydrone is an equimolar mixture of quinone and hydroquinone. In an aqueous solution of qunihydrone, the following reversible reaction takes place

$$\text{Q} \quad + 2H^+ + 2e^- \rightleftharpoons \quad \text{QH}_2$$
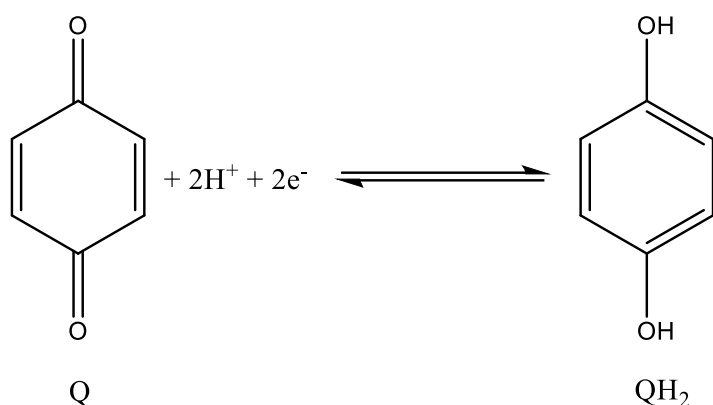
Because the above reversible reaction involves H ions, the quinhydrone in solution function as redox electrode in contact with an inert conductor such as platinum. The potential of the quinhydrone electrode is sensitive to the pH of the solution.

The EMF of the cell is given as

$$E_{cell} = E_Q - E_{SCE}$$
$$E_Q = E^O_Q - (2.303RT/F) \log (1/H^+)$$
$$E_{cell} = E^O_Q - (2.303RT/F) \log (1/H^+) - 0.242$$
$$E_{cell} = E^O_Q - (2.303RT/F) \text{ pH} - 0.242$$
$$E_{cell} = 0.699 + 0.0591 \log (H^+) - 0.242$$
$$E_{cell} = 0.457 + 0.0591 \log (H^+)$$
$$(\text{or})$$
$$E_{cell} = 0.457 - 0.0591 \text{ pH}$$

As the titration proceeds, H ion concentration decreases and hence EMF of the cell decrease slowly but in the vicinity of the equivalence point the rate of change of potential is very rapid or maximum. On crossing the equivalence point, again EMF changes in small decrements. From the sharp break in the curve, equivalence point can be determined, from which the strength of the acid can be calculated.

Determination of dissociation constant of weak acid:

The Henderson-Hasselbalch equation is

$$pH = pKa + log \frac{[salt]}{[acid]}$$

At half neutralization point, the concentrations of the salt and acid are equal,

Then $log \frac{[salt]}{[acid]} = 1$; $\qquad\qquad$ **pH = pKa**

A. K. Singh, P. Shriwas, GGV, Bilaspur (C.G.)

$$\frac{(0.457 - Ecell)}{0.0591} = pKa$$

Ka = antilog ( )

Procedure: Take 20 mL of 0.1M AcOH in a clean 100 mL beaker and add sufficient amount of distilled water (30 mL) so that the electrodes are completely dipped. Add a pinch of quinhydrone to saturate the solution and dip indicator (working) electrode in the solution. Combine the Pt electrode (contact electrode) with the calomel electrode through a salt bridge. The two electrodes are connected to the potentiometer. Once the potentiometer is standardized, add 1mL of 0.1M NaOH from the micro burette to acetic acid solution taken in a beaker. Stir the solution carefully and note down the corresponding EMF value. Continue the addition of NaOH solution from the burette and note the EMF and tabulate the data.

Model Tabular Form:

S. No.	Volume of NaOH (mL)	EMF (mv)	ΔE (mv)	ΔV (mL)	ΔE/ΔV
1.	0				
2.	1				
3.	2				
4.	3				
5.	4				
6.	5				

Graphs:
1) Plot a graph between EMF and volume of NaOH This gives an equivalence point (sigmoid)
2) Plot a graph between ΔE/ΔV and volume of NaOH A differential graph is obtained.

A. K. Singh, P. Shriwas, GGV, Bilaspur (C.G.)

Sigmoid curve	First derivative curve
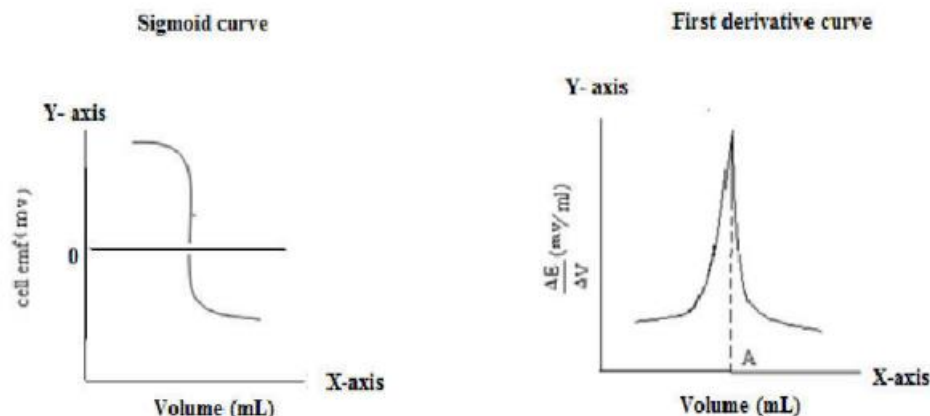
Result: The end point for the titration of 20mL AcOH against 0.1M NaOH is _____mL.

Strength/Concentration of given AcOH solution is _____M

Dissociation constant (K) for weak acid = _____

Precautions:

1. Always wear appropriate eye protection (i.e., chemical splash goggles) in the laboratory.
2. Wear disposable gloves, when handling hazardous materials.
3. Wear a full-length, long-sleeved laboratory coat or chemical-resistant apron.
4. Wear shoes that adequately cover the whole foot; low-heeled shoes with non-slip soles are preferable.
5. Clean apparatus before and after use.

4. **Determination of Single Electrode Potential.**

Aim: To determine the single (standard) electrode potential of silver electrode Potentiometrically.

Requirements

Apparatus: Potentiometer, calomel electrode, silver electrode, salt bridge, beaker.

Chemicals: 0.1M AgNO$_3$, saturated KCl etc.

Principle: To measure changes, the indicator electrode is coupled with a reference electrode using a salt bridge. The potential of reference electrode remains unchanged during the progress of reaction.

The cell set up is as follows
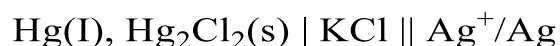
$$Hg(I), Hg_2Cl_2(s) \mid KCl \parallel Ag^+/Ag$$
$$E_{Ag} = E^O_{Ag} - (2.303RT/F) \log (1/Ag^+)$$
$$E_{cell} = E_{Ag} - E_{SCE}$$
$$E_{Ag} = E^O_{Ag} - (2.303RT/F) \log (1/Ag^+)$$
$$E_{cell} = E^O_{Ag} - (2.303RT/F) \log (1/Ag^+) - 0.242$$
$$E_{cell} = E^O_{Ag} + 0.0591 \log (Ag^+) - 0.242$$
$$E^O_{Ag} = E_{cell} - 0.0591 \log (Ag^+) + 0.242$$

A. K. Singh, P. Shriwas, GGV, Bilaspur (C.G.)

Procedure: Take 20ml of 0.1M AgNO₃ into a 100ml beaker and dip the Ag electrode in the beaker and connect it to the Potentiometer. Measure the potential difference of the solution. Remove 10ml of solution from the beaker and add 10ml of distilled water such that the solution is diluted to M/20. Now record the EMF value of the cell. Repeat the process with various concentration of AgNO₃ say M/40, M/80, M/160. E^O_{Ag} can be calculated using Nernst equation.

Model tabular form:

S. No.	Conc. Of AgNO₃	E_{cell} (Volt)	log [Ag⁺]	$E^O_{Ag} = E_{cell} - 0.0591 \log (Ag^+) + 0.242$
1.	0.1M			
2.	0.05M			
3.	0.025M			
4.	0.0125M			
5.	0.00625M			

Graph: Plot a graph between E_{cell} (EMF) and log [Ag⁺]. Intercept is equal to E^O_{Ag} 0.242. From this E^O_{Ag} can be determined

$$\text{Intercept} = E^0_{Ag} - 0.242$$
$$E^0_{Ag} = \text{Intercept} + 0.242$$

$$\text{Intercept} = E^0_{Ag} - 0.242$$
$$E^0_{Ag} = \text{Intercept} + 0.242$$

A. K. Singh, P. Shriwas, GGV, Bilaspur (C.G.)
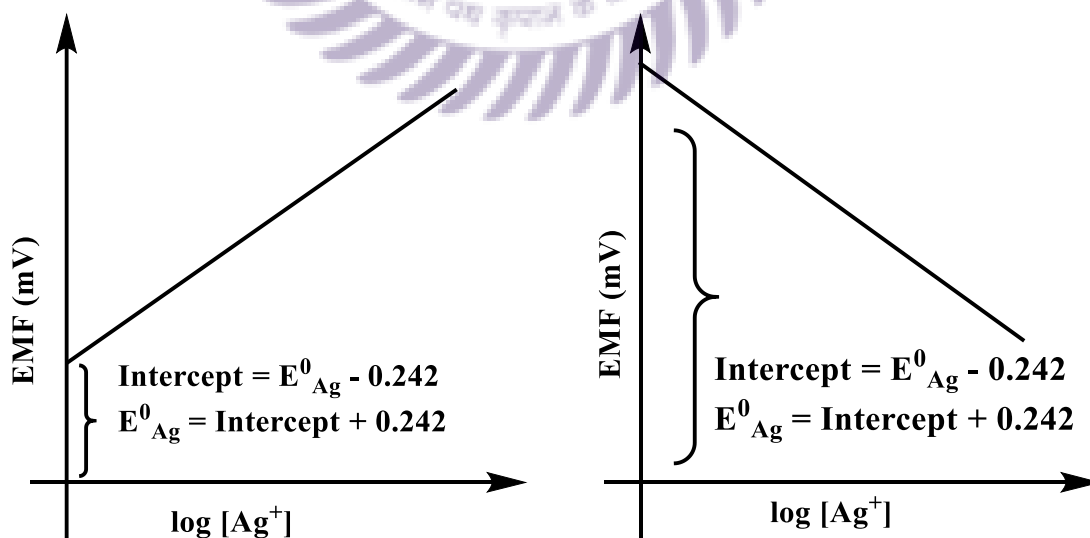
Result:

E^O_{Ag} (From graph) = _____ Volts

E^O_{Ag} (From Calculations) _____ Volts

Precautions:
1. Always wear appropriate eye protection (i.e., chemical splash goggles) in the laboratory.
2. Wear disposable gloves, when handling hazardous materials.
3. Wear a full-length, long-sleeved laboratory coat or chemical-resistant apron.
4. Wear shoes that adequately cover the whole foot; low-heeled shoes with non-slip soles are preferable.
5. Clean apparatus before and after use.

5. Adsorption of acetic acid on animal charcoal.

Aim: To verify the adsorption isotherm for acetic acid adsorption on animal charcoal.

Requirements
Apparatus: Reagent bottles-5, burette, funnel, watmann paper no. 41, pipette, conical flask.

Chemicals: 0.1N acetic acid, 0.1N NaOH, charcoal 1gm, phenolphthalein.

Principle: Freundlich proposed a relation between the amount of solute adsorbed on a definite amount adsorbent and the equilibrium concentration of the adsorbate in the solution. According to which

$$x/m = kC_e^{1/n}$$

$$\log(x/m) = \log k + 1/n \log C_e$$

where, x = amount of solute adsorbed;
m = mass of the adsorbent;
C_e = equilibrium concentration of adsorbate;
k = constant.
n = no. of layers of acetic acid adsorbed to the surface layer of charcoal.

Procedure: Take 5 clean reagent and number them 1-5. Prepare the following solution mixtures.
1. Stopper each bottle after adding 1gm of charcoal and shake the bottles in a rotatory motion and allow them to stand for at least 1 hour.

2. Fill the burette with 0.1N NaOH solution. Pipette out 10ml of stock 0.1M acetic acid solution into a clean conical flask and add 1 or 2 drops of phenolphthalein indicator and titrate it against sodium hydroxide solution. The end point is noted when a faint pink colour is observed.

3. After 1 hour filter the contents of each bottle separately through a watmann filter

paper no. 41. while filtering rejects the first 5ml of the filtrate and collect the rest. Wait until the filtration is complete and pipette out 10ml of the filtrate in to a clean conical flask and titrate it against with 0.1N NaOH by adding 1-2 drops of phenolphthalein indicator. The end point is noted when a faint pink colour is observed.

4. Repeat the procedure with the filtrate of other bottles and tabulate the results.

Observation Table:

Table 1

Bottle No.	Volume of Acetic acid	Water
1.	50ml	0ml
2.	40ml	10ml
3.	30ml	20ml
4.	20ml	30ml
5.	10ml	40ml

Table 2

Bottle No.	Initial concentration (C_i)	Volume of N/10 NaOH (C_e)	$x = (C_i - C_e) / 20$	$\log (x/m)$	$\log (C_e)$
1.					
2.					
3.					
4.					
5.					

Graph: A graph is plotted by taking $\log(x/m)$ on y-axis and $\log(C_e)$ on x-axis. A straight-line cutting y-axis is obtained. The slope of the straight line is equal to $1/n$ and the intercept is equal to $\log(k)$.

A. K. Singh, P. Shriwas, GGV, Bilaspur (C.G.)

The graph shows $\log (x/m)$ on the y-axis versus $\log (C_e)$ on the x-axis, with slope = $1/n < 1$ and intercept $\log(k)$.
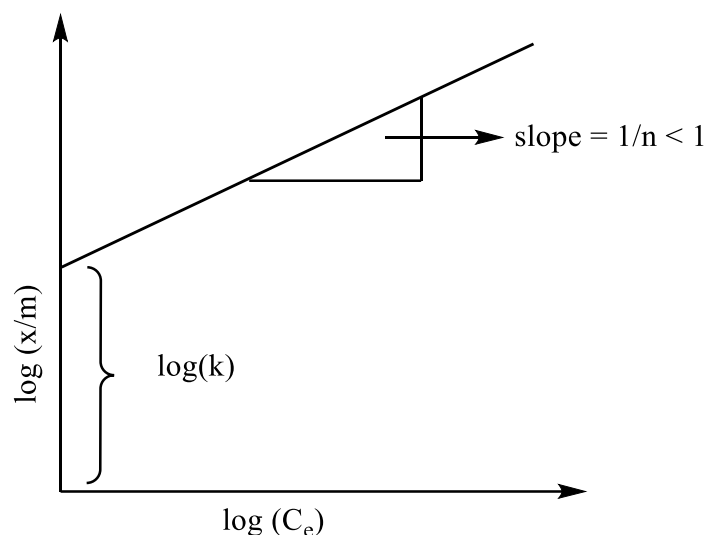
Result: The Freundlich adsorption isotherm is verified.

Precautions:

1. Always wear appropriate eye protection (i.e., chemical splash goggles) in the laboratory.
2. Wear disposable gloves, when handling hazardous materials.
3. Wear a full-length, long-sleeved laboratory coat or chemical-resistant apron.
4. Wear shoes that adequately cover the whole foot; low-heeled shoes with non-slip soles are preferable.
5. Clean apparatus before and after use.

6. Study of effect of added electrolyte on CST of phenol water system.

Aim: To study of effect of added electrolyte on CST of phenol water system.

Requirements

Apparatus: Boiling tube, beaker, two holed rubber cork thermometer, wire stirrer.

Chemicals: Phenol, 1%NaCl, distilled water.

Principle: Certain mixtures are partially miscible and are soluble in each other only under certain conditions. When the temperature is altered at constant pressure the initially partially miscible mixture become completely miscible and the temp at which they become completely miscible is known as CST. The CST of a solution is specific and is sensitive to the impurities.

If an impurity is soluble only in one of the components it alters the CST of the system. Addition of impurities to the phenol water system raises the CST of the system. The change in CST is found to be linearly proportioned to the % of impurity.

Procedure: Prepare 100ml of 1% NaCl solution. Fix the thermometer and wire stirrer into the two holed rubber stopper. Now prepare the following compositions.

A. K. Singh, P. Shriwas, GGV, Bilaspur (C.G.)

Volume of phenol	5	5	5	5	5
Volume of 1% NaCl	10	8	6	4	2
Volume of H_2O	0	2	4	6	8

The above compositions are taken in a boiling tube which is stoppered with rubber cork containing thermometer and wire stirrer. The boiling tube is placed in a beaker containing water. Increase the temperature of the system by heating it. Stir the mixture constantly with the help of wire stirrer and it appears to be cloudy. The liquid mixture is heated until the last trace of cloudiness disappears. The temperature of the system is noted as heating temperature (T_1°c). Now immediately remove the burner and allow it to cool and the temperature at which the cloudiness reappears is noted as cooling temperature as (T_2°c) and the mean of the two temperatures is noted. The above procedure is repeated for all the other compositions and these heating and cooling temperatures are noted.

Graph: Plot a graph between the percentage of salt in solution on x-axis and its miscibility temperature on y-axis. A straight line passing through origin is obtained.
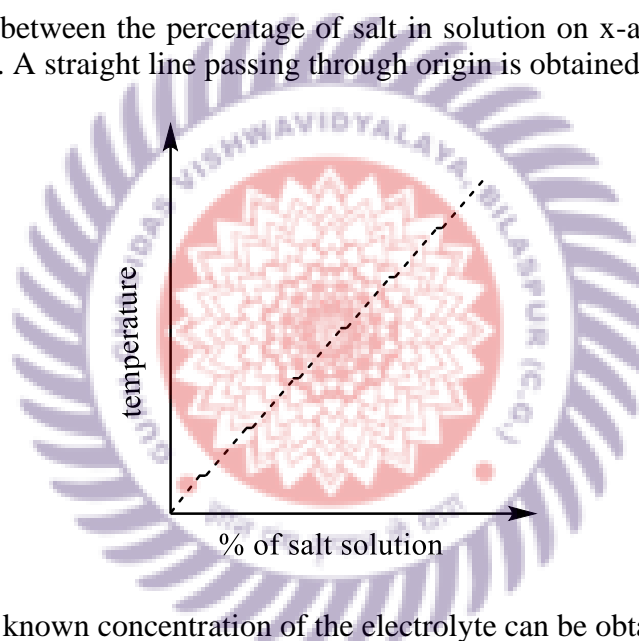
Result: Using this un known concentration of the electrolyte can be obtained.

Precautions:

1. Always wear appropriate eye protection (i.e., chemical splash goggles) in the laboratory.
2. Wear disposable gloves, when handling hazardous materials.
3. Wear a full-length, long-sleeved laboratory coat or chemical-resistant apron.
4. Wear shoes that adequately cover the whole foot; low-heeled shoes with non-slip soles are preferable.
5. Clean apparatus before and after use.

A. K. Singh, P. Shriwas, GGV, Bilaspur (C.G.)

7. Determination of CST of solution.

Aim: To determine the critical solution temperature of the phenol water system.

Requirements
Apparatus: Boiling tube, beaker, wire stirrer thermometer, two holed rubber cork, graduated pipettes

**Chemicals:** Phenol, 1%NaCl, distilled water.

Principle: Certain solution mixtures are partially miscible. The partially miscible liquid pairs are soluble in each other only up to certain limits. On addition of small quantity of either of the liquid increases the relative immiscibility beyond a certain temperature the two liquids are completely miscible. This temperature at which the two partially miscible liquids become completely miscible is known as critical solution temperature.

Procedure: Take a clean beaker filled with tap water place it on the bunsen burner. Place the thermometer and wire stirrer into the boiling tube using a two holed rubber stopper. Now prepare the following mixtures.

Vol. of phenol	9ml	8ml	7ml	6ml	5ml	4ml	3ml	2ml	1ml
Vol. of water	1ml	2ml	3ml	4ml	5ml	6ml	7ml	8ml	9ml

The above compositions are taken in a boiling tube which is stoppered with rubber cork containing thermometer and wire stirrer. The boiling tube is placed in the beaker containing water. The temperature of the system is increased by heating the water bath. The liquid mixture is constantly stirred with the help of wire and the liquid mixture appears to be cloud. The liquid mixture is heated until the last trace of cloudiness disappears. The temperatures of the mixture are noted as heating temperature (T_1^o c) and immediately remove the burner and allow it to cool and the temperature at which the cloudiness just appears is also noted as (T_2^o c). The above procedure is repeated for all the other compositions and their heating and cooling temperatures are noted.

Graph: Plot a graph by taking composition of phenol on x-axis and mean of the temperatures on y-axis. A parabolic curve is obtained. The value of maximum point on x-axis gives the composition and that on y-axis gives the temperature at which the two liquid mixtures remain completely miscible.

Composition of phenol in H_2O
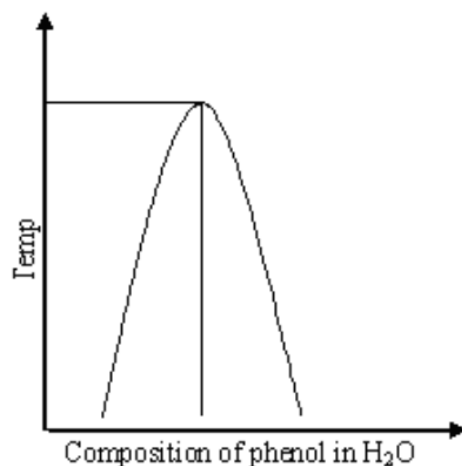
Result: CST of phenol in water = 60^oc

Composition of phenol =

Precautions:

1. The volume of solution of the solution should be taken in such a way that the bulb of thermometer is completely dipped into the mixture.
2. Always wear appropriate eye protection (i.e., chemical splash goggles) in the laboratory.
3. Wear disposable gloves, when handling hazardous materials.
4. Wear a full-length, long-sleeved laboratory coat or chemical-resistant apron.
5. Wear shoes that adequately cover the whole foot; low-heeled shoes with non-slip soles are preferable.
6. Clean apparatus before and after use.

8. Chemical Kinetics

1. Acid-catalyzed hydrolysis of Methyl acetate.

Aim: To Study the Acid catalyzed hydrolysis of an ester (Methyl Acetate) under Pseudo conditions (first order reaction) and determine the rate constant (k).

Requirements

Apparatus: Burette, Pipette, Conical flask, Reagent bottles, etc.

Chemicals: Methyl Acetate, HCl (1M/2M), NaOH (0.5M), Phenolphthalein indicator, crushed ice.

Principle: Hydrolysis of an ester in aqueous medium is very slow. Hence the reaction rate is enhanced by an acid (HCl). The reaction is as follows

A. K. Singh, P. Shriwas, GGV, Bilaspur (C.G.)

Methyl acetate $+$ H$_2$O $\xrightleftharpoons{\text{H+}}$ Acetic acid $+$ MeOH
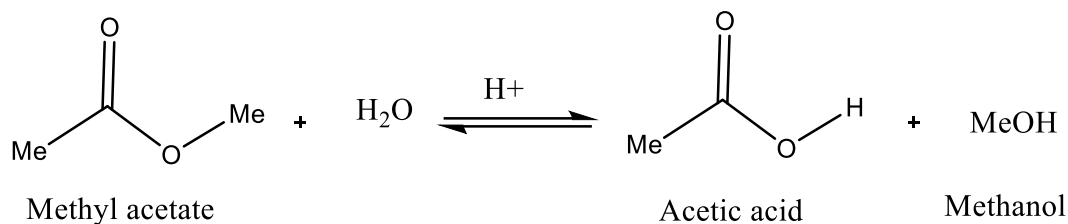
$$\text{Rate} = k^1[\text{Ester}]\,[\text{H}_2\text{O}]$$

$$\text{Rate} = k\,[\text{Ester}]^1 \ (\text{where } k = k^1\,[\text{H}_2\text{O}])$$

$$k = \text{Pseudo first order rate constant}$$

The reaction is of 1st order with respect to Methyl Acetate, since the concentration of water is taken in large excess and hence the change in water concentration is very less or virtually constant during the course of the reaction. Hence the reaction is referred as Pseudo first order reaction.
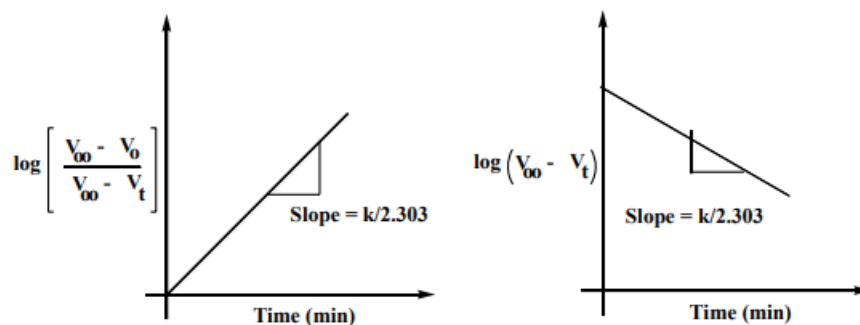
Rate constant,

$$k = \frac{2.303}{t} \log \left[\frac{V_\infty - V_0}{V_\infty - V_t} \right] \ \text{min}^{-1}$$

Procedure: Take 100 ml of 1M HCl in a clean reagent bottle and fill the burette with 0.5MNaOH solution. Now add 10 ml of pure Methyl Acetate to the reagent bottle containing HCl and shake the solution. Immediately (within 10-15 seconds), pipette out 10 ml of the reaction mixture into a clean conical flask containing ice cold water in order to quench the reaction. Now add 2 or 3 drops of Phenolphthalein indicator and titrate against NaOH solution. Note down the titre value in the tabular form as Vo. Repeat the same titration procedure for every 10 minutes of regular intervals of time by doing up to 60 minutes. Tabulate the titre values as Vt.

For V∞: Heat the remaining reaction mixture for half an hour by maintaining 50 – 60oC. Cool it to room temperature under tap water. Pipette out 10 ml of the above reaction mixture into a clean conical flask (without ice water) and titrate against NaOH solution using phenolphthalein indicator. Take the titre value as V∞.

Repeat the same experimental procedure for 2M HCl solution and tabulate the data

Model Graphs: Plot a graph between $\log \left[\frac{V_\infty - V_0}{V_\infty - V_t} \right]$ and time, straight line passing through origin is obtained. From the slope rate constant (k) can be calculated. Plot another graph between log (V∞-Vt) and time, a straight line with negative slope is obtained.

A. K. Singh, P. Shriwas, GGV, Bilaspur (C.G.)

Left graph: $\log\left[\dfrac{V_{\infty} - V_0}{V_{\infty} - V_t}\right]$ vs Time (min), Slope = k/2.303

Right graph: $\log\left(V_{\infty} - V_t\right)$ vs Time (min), Slope = k/2.303

Model tubular form:

S. No.	Time (min)	Volume of NaOH (ml)	$(V_{\infty} - V_t)$	$\log (V_{\infty} - V_t)$	$[V_{\infty} - V_0 / V_{\infty} - V_t]$	$\log [V_{\infty} - V_0 / V_{\infty} - V_t]$	$k = 2.303/t$ $\log [V_{\infty} - V_0 / V_{\infty} - V_t]$
1.	0						
2.	10						
3.	20						
4.	30						
5.	40						
6.	50						
7.	60						
8.	V_{∞}						

Result: The reaction is of Pseudo first order.

Rate constant from the experiment data = ……………………… min^{-1}

Rate constant from the graph - 1 = …………………………. min^{-1}

Rate constant from the graph - 2 = …………………………… min^{-1}

Precautions:

1. Always wear appropriate eye protection (i.e., chemical splash goggles) in the laboratory.
2. Wear disposable gloves, when handling hazardous materials.
3. Wear a full-length, long-sleeved laboratory coat or chemical-resistant apron.
4. Wear shoes that adequately cover the whole foot; low-heeled shoes with non-slip soles are preferable.
5. Clean apparatus before and after use.

A. K. Singh, P. Shriwas, GGV, Bilaspur (C.G.)

2. Peroxydisulphate- Iodide reaction.
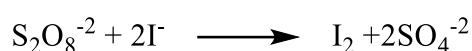## Determination of overall order rate constant of a reaction.

Aim: To verify the order and determine the rate constant by following the kinetics of potassium Persulphate and Potassium iodide reaction volumetrically.

Requirements
Apparatus: Iodination flask, burette, pipette, standard flask, conical flask, beaker, beaker, measuring jar, stop watch etc.

Chemicals: 0.1M KI, 0.05M $K_2S_2O_8$, M/200 hypo, Ice cold water, starch indicator.

Chemical reaction:

$$S_2O_8^{-2} + 2I^- \longrightarrow I_2 + 2SO_4^{-2}$$

$$2Na_2S_2O_3 + I_2 \longrightarrow 2NaI + Na_2S_4O_6$$

Principle and Mechanism:

$$S_2O_8^{-2} + I^- \longrightarrow (S_2O_8I)^{-3}$$

$$(S_2O_8I)^{-3} + I^- \longrightarrow 2SO_4^{2-} + I_2$$

Adding the above two equations,

$$2I^- + S_2O_8^{-2} \longrightarrow 2SO_4^{2-} + I_2$$

This is a reaction of the second order as the concentrations of both the reactants appear in the rate equation to first power. During the reaction, iodine is liberated and progress of the reaction is observed by titrating the liberated iodine against sodium thiosulphate solution (hypo) at regular intervals of time. The titre values are proportional to iodine formed and therefore to the amount of peroxo disulphate which has disappeared. A known quantity of $K_2S_2O_8$ is mixed with definite known quantity of KI. Let the initial concentration of $K_2S_2O_8$ be 'a' moles per litre; 'x' be the concentration of I_2 formed in the reaction at any time,'t'. Vt is the volume of hypo required at any time 't' and V∞ is the volume of hypo required at the end of the reaction. Then the reaction follows second order and the rate law is given by:

$$\text{Rate} = k_2 \, [KI] \, [K_2S_2O_8]$$

$$\frac{x}{x(a-x)} = \left[\frac{1}{(a-x)} - \frac{1}{a} \right] = k_2 t$$

A. K. Singh, P. Shriwas, GGV, Bilaspur (C.G.)

Which may be expressed in terms of volumes V_t and V_∞ as:

$$\frac{V_t}{V_\infty (V_\infty - V_t)} = k_2 t$$

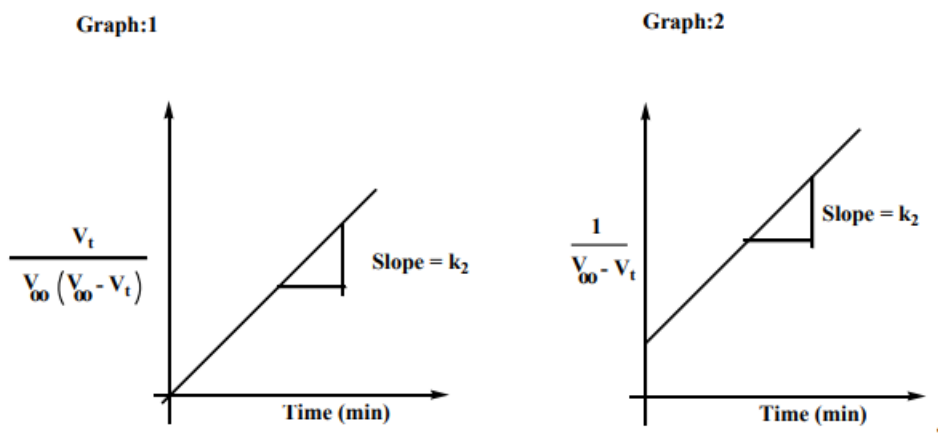$$k_2 = \frac{1}{t} \left[\frac{V_t}{V_\infty (V_\infty - V_t)} \right]$$

Procedure: Take 50ml of 0.05M K2S2O8 into an iodination flask. Fill the burette with M/200 hypo. Keep a conical flask ready for titration, with some ice-cold water and a few ice pieces in it. Now measure 50ml of 0.1M KI, add it to the iodination flask containing K2S2O8 and immediately note down the time. After 10 minutes pipette out 10ml of reaction mixture into a conical flask containing ice cold water and add few drops of freshly prepared starch solution. The solution turns blue. The end point is indicated by the disappearance of blue colour. Note down the titre value, Vt for 10 minutes. And repeat the similar procedure and determine Vt corresponding to the times 20, 30, 40 …. minutes respectively.

For V∞ value: At the early stage of the reaction i.e., after 2 to 3 minutes, pipette out 10ml of the reaction mixture into a clean conical flask and add excess of KI to it (one spatula of solid KI) cover it with watch glass and keep it in dark for about 30 minutes. After 30 minutes take out the flask and wash the lid of watch glass into the conical flask. Titrate the contents of the conical flask against hypo and note down the reading as V∞.

Model tabular form:

Time (min)	Volume of hypo required (ml)	(V∞- Vt)	1 / (V∞- Vt)	V_t / V_∞ (V∞- Vt)	k = 1/t [V_t / V_∞ (V∞- Vt)]
0					
10					
20					
30					
40					
50					
60					
∞	**V∞ =**				

A. K. Singh, P. Shriwas, GGV, Bilaspur (C.G.)

Model graph: Plot a graph between $V_t / V_\infty(V_\infty - V_t)$ Vs time, Straight line passing through origin is obtained, slope of the line is equal to k_2. Draw another plot between $1/(V_\infty - V_t)$ Vs time, a straight line with positive slope and intercept is obtained.

Graph:1

Graph:2

$$\frac{V_t}{V_\infty (V_\infty - V_t)}$$

Slope = k_2

$$\frac{1}{V_\infty - V_t}$$

Slope = k_2

Time (min)

Time (min)

Result:

k$_2$ value from calculations = …………… lit/mole/min

From Graph – 1, k_2 =

From Graph –2, k_2 =

Precautions:

1. Always wear appropriate eye protection (i.e., chemical splash goggles) in the laboratory.
2. Wear disposable gloves, when handling hazardous materials.
3. Wear a full-length, long-sleeved laboratory coat or chemical-resistant apron.
4. Wear shoes that adequately cover the whole foot; low-heeled shoes with non-slip soles are preferable.
5. Clean apparatus before and after use.