MUFFAKHAM JAH COLLEGE

OF
ENGINEERING AND TECHNOLOGY

(SULTAN-UL-ULOOM EDUCATION SOCIETY)

BANJARA HILLS, HYDERABAD - 500 034.

A MANUAL OF CHEMISTRY PRACTICALS

FOR B.E. I YEAR SEMESTER-II
ACADEMIC YEAR 2017-2018

VOLUME II : INSTRUMENTAL CHEMICAL ANALYSIS

Name: _____

Branch (section): _____

Roll No. 1604 - 17 - _____

MJCET

Department of Chemistry

List of Staff members

Teaching

Sl.No.	Name	Designation
1.	Dr. Y. Arun Kumar	Professor & Head
2.	Dr. Aneesa Ferdose	Assistant Professor
3.	Mrs. Amina Sarwar	Assistant Professor
4.	Dr. V. Shanthi	Assistant Professor
5.	Dr. MGV Satyanarayana	Assistant Professor
6.	Dr. Sariah Sana	Assistant Professor
7.	Mrs. Syeda Arshiya Banu	Assistant Professor
8.	Dr. J. Pragathi	Assistant Professor
9.	Dr. Soghra Hyder	Assistant Professor

Non-Teaching

Sl.No.	Name	Designation
1.	Mr. Rashed Ali	Lab Assistant
2.	Mr. Misba ur Rahman	Lab Assistant
3.	Mr. Syed Mansoor ul Hussain	Lab Attendant
4.	Mr. llyas Pasha	Lab Attendant
5.	Mr. Syed Zameeruddin	Lab Attendant

CONTENTS

INSTRUMENTAL CHEMICAL ANALYSIS

Safety instructions to the students

1) **Poisonous Chemicals:** All of the chemicals have some degree of health hazard. *Never taste* any chemical in the laboratory unless specifically directed to do so. Avoid breathing toxic vapours. When working with volatile chemicals, strong acid and bases, using ventilating hoods. If you are asked to taste the odour of a substance, do it by *wafting* a bit of the vapour towards your nose. Do not stick your nose in and inhale vapour directly from the test tube. Always wash your hand before leaving the laboratory.

2) **Cut and Burns:** Use glassware with care, Glassware is breakable and may cause cuts. When a piece of glass is heated, it gets hot very quickly. Since hot glass looks just like a cold one, handle it with a tong. Do not use any cracked or broken glass equipment. It may ruin an experiment and worse, it may cause serious injury. Place it in a waste glass container. Do not throw them into the wastepaper container or regular waste container.

3) **Eating and drinking:** Any type of food is prohibited in the laboratory at all times and smoking is not allowed.

4) **Clothing and Footwear:** Everyone must wear a lab coat in the lab. Long hair should be securely tied back to avoid the risk of setting it on fire. If large amount of chemicals are spilled on your body, immediately remove the contaminated clothing and use the safety shower if available. Make sure to inform your instructor about the problem. Do not leave your coats and back packs on the bench. No Cell Phones, headphones and Walkman are allowed in the lab because they interfere with your ability to hear what is going on in the Lab.

5) **Eye Protection:** Because the eyes are particularly susceptible to permanent damage by corrosive chemical as well as flying objects, preferably wear safety goggles in the Laboratory. No sunglasses are allowed in the laboratory. Contact lenses have potential hazard because the chemical vapours dissolve in the liquids covering the eye concentrate behind the lenses. If you are using contact lenses consult with your instructor. If possible try to wear prescribed glasses under your safety goggles. In case of any accident that a chemical splashes near your eyes, immediately wash your eyes with a lot of water and inform your instructor. Especially, when heating a test tube do not point its mouth to anyone or you.

6) **Fire:** in case of fire or accident, inform your instructor immediately. Note the location of fire extinguishers as soon as you enter the laboratory so that you may use them if needed. Never perform an unauthorized experiment in the laboratory.

INSTRUMENTAL CHEMICAL ANALYSIS

Introduction :

Quantitative chemical analysis can also be done with the help of various analytical instruments like Colorimeter, Conductometer, PH meter, Potentiometer and Spectrometer.

<u>Advantages of Instrumental analysis:</u>

- Low concentration of sample required.

- Highly sensitive and selective

- Reliable measurements.

- Fast determination.

- Indicators are not required.

Method	Instrument	Phenomenon underlying the method	Quantity measured
Optical methods			
1. Colorimetry	1.colorimeter	Interaction of matter with Electromagnetic radiation	Absorbance Or Transmittance
2. SpectroPhoto-metry	2.Spectrophoto-meter		
Electrochemical methods		Changes during chemical reactions	
1.Conductometry.	1.Conductometer	Conductivity of the solution	Conductance
2.pH metry	2.pH meter	H^+ ion concentration of the solution	pH
3.potentiometry	3.Potentiometer	Electrode potential difference of the cell	Emf

CONDUCTOMETRY

The electrical conduction in electrolytes is due to flow/movement of the ions followed by their electrode reaction at the anode and cathode respectively. This amounts to indirect transfer of alter electrons through the electrolyte (conduction in liquids) as free electrons cannot exist in solution.

During the measurement of conductivity of the solution, the concentration of electrolyte (C) and the applied e.m.f. (E) must be constant. This is possible by using alternate current (A/C) of high frequency and coating the platinum electrodes with platinum black (to prevent the back e.m.f.).

CONDUCTIVITY CELL:

Conductivity cell is a special type of cell used for measuring the conductance of an electrolytic solution. The cell consists of two electrodes. Each electrode is a platinum disc or plate coated with finely divided platinum black. The electrode is connected to a platinum wire which is fused to a glass tube. The glass tubes are firmly fixed in the cell so that the distance between the electrodes would not change during the experiment. The cell constant is fixed for the conductivity cell. The cell is open at one end. An aqueous solution of the electrolyte whose conductance has to be measured is placed in a beaker. The conductivity cell is kept in the solution and is connected to the conductivity meter.

*There are no foolish questions, & no man becomes a fool until he has stopped asking questions.

CELL CONSTANT:

The cell constant (x) of a conductivity cell is the ratio of the distance between the two electrodes to the area of cross-section of each electrode.

$$X = \text{Cell Constant} = \frac{\text{Distance between the electrodes}}{\text{Cross}-\text{sectional area of electrode}} = \frac{l}{a}$$

Units of cell constant = cm^{-1}; m^{-1}

SPECIFIC CONDUCTIVITY (K):

Specific conductivity is the conductivity by the ions present in one cubic centimeter of the solution. If the cell constant of a conductivity cell is 1.0, the measured conductivity of the electrolyte will also be its specific conductivity. The specific conductivity of an electrolyte is expressed in ohm^{-1} cm^{-1}, mho cm^{-1}, $S.cm^{-1}$, $S.m^{-1}$.

$$\text{Cell Constant} = \frac{\text{Specific conductance of KCl}}{\text{Measured conductance of KCl}}$$

*The important thing is not to stop questioning

- Einstein.

CONDUCTOMETRIC ACID – BASE TITRATIONS

THEORY:

The electrical conductance of an electrolytic solution depends on :

i)   Number of ions present in solution i.e., ionic concentration.

ii)   The ionic mobilities (size of ions)

iii)   Temperature of the solution

iv)   Nature of electrolyte

v)   Dilution of solution

The ionic mobilities of various ions in aqueous solution at 25^oC are generally in the range. 4.8×10^{-4} cm sec^{-1} under potential of 1 Volt cm^{-1}. But the ionic mobilities of H$^+$ and OH$^-$ are abnormally high (H$^+$ = 36.8×10^{-4}, OH$^-$=20.5 x 10^{-4} cm2 sec^{-1} Volt^{-1}). Thus the conductance of an electrolyte is quite sensitive to the concentration and ionic mobilities in acid-base titrations of H$^+$ and OH$^-$ ions.

In the conductometric acid-base titrations the course of the titration as base is added to the base, is followed from the gradual change in the conductivity of the titrand.

1. **Titration of Strong Acid (HCl) with Strong Base (NaOH)**

    In a solution HCl is completely ionized. The mobility of H$^+$ ions is high, hence the conductance of HCl solution will also be high. As NaOH is added to HCl, the fast moving H$^+$ ions are removed by OH$^-$ ions of the base as water.

    H$^+$ + Cl$^-$ + Na$^+$ + OH$^-$ $\rightarrow$ Na$^+$ + Cl$^-$ + H$_2$O (Undissociated)
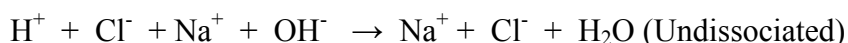
In solution water is little ionized and Na^+ ions added are slow moving compared to H^+ ions of the acid. Hence, as the titration proceeds, the conductance of the titrand (HCl) decreases gradually till the equivalence point is reached. At equivalence point the conductance of HCl will be minimum. Addition of the base beyond the equivalence point results in a gradual increase in conductance mainly due to the fast moving OH^- ions. Model graph for titration of (A) Strong acid Vs Strong base and (B)Weak acid Vs Strong base is given below.

(A)

(B)
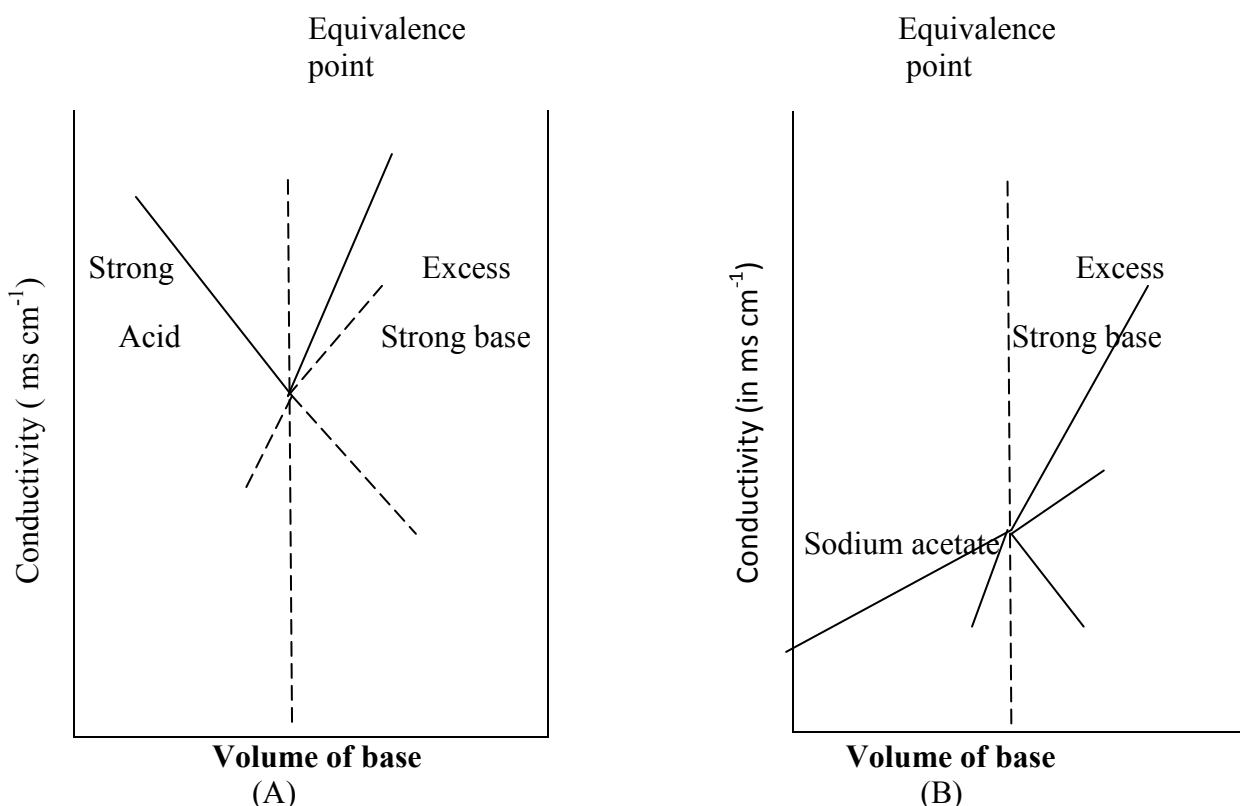
The plot of the conductance of the solution against the volume of the titrant (base) added gives two straight lines. The point of intersection of the intrapolated lines is the equivalence point of the titration.

2. Titration of Weak Acid (CH₂COOH) with Strong Base (NaOH)

The conductance of an aqueous solution of acetic acid will be low, because of the low concentration of H^+ ions, as acetic acid is feebly ionized. As NaOH is added, **highly ionized sodium acetate** is formed and effectively the net result of the titration is the replacement of unionized CH_3COOH molecules by the highly ionized sodium acetate. Therefore there will be a gradual increase in the conductance.

$$CH_3COOH + Na^+ + OH^- \rightleftharpoons CH_3COO^- + Na^+ + H_2O$$

(aq)   (aq)   (aq)   (aq)   (aq)
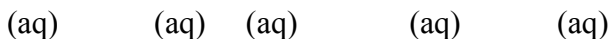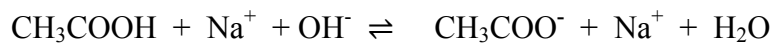
After the equivalence point, any further addition of NaOH will produce a more rapid increase in conductance due to highly mobile OH^- ions. The point of intersection of the lines, in the plot of the conductance against the volume of the alkali added, is the end point.

3. **Titration of a mixture of strong and weak acids**

   (HCl + CH$_3$COOH) against strong base (NaOH)

   In the mixture of acids, the H^+ ion concentration is almost exclusively from HCl. When the base is added HCl will react first, as indicated by a gradual decrease in conductance. When all the HCl is used up, CH$_3$COOH starts reacting, leading to a gradual increase in conductance. Beyond the equivalence point with the addition of excess NaOH conductance will shoot up. The Plot of the conductance Vs the volume of alkali added will show two independent end points on the graph, first one is equivalence point for the end point of HCl Vs NaOH and the second one for the end point of HCl + CH$_3$COOH Vs NaOH.

   V_1 = Volume of NaOH required for neutralization of HCl.

   $(V_2 - V_1)$ = Vol. of NaOH required for neutralization of CH$_3$COOH
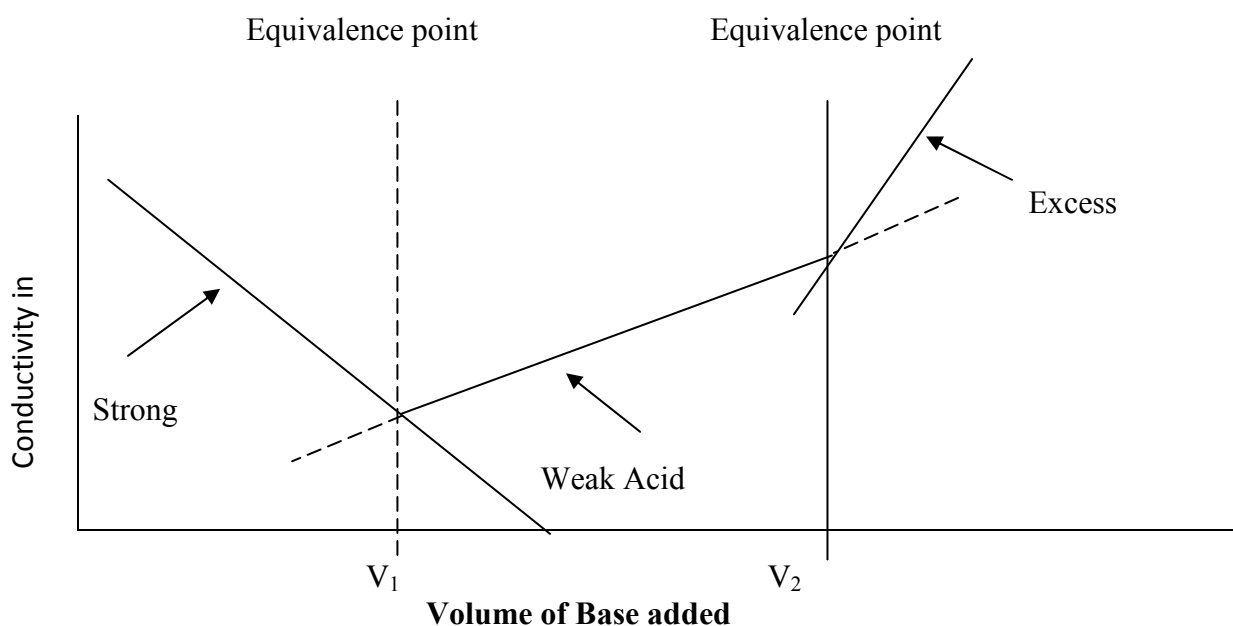
Procedure:

Preparation of Std. Oxalic Acid Solution:

Weigh about 0.63 g. of oxalic acid, in a weighing bottle. Transfer it into a clean 100 ml std. flask through funnel. Dissolve it in distilled water and make a homogeneous solution upto the mark. From the wt. of Oxalic acid calculate the normality of std solution.

A. Standardisation of NaOH (link) solution:

Pipette out 20 ml of the oxalic acid solution into a clean conical flask. Add 2-3 drops of phenolphthalein indicator. Titrate the contents of the flask till a light pink colour appears. Repeat the titration to get two concordant titre values.

Requirements: Conductivity meter, conductivity cell, burette, pipette, beakers.

Chemicals:

HCl	:	~0.05N (N/20)	
CH₃COOH	:	~0.05N (N/20)	
NaOH	:	~0.5N (N/2)	

Chemicals: HCl   :   ~0.05N ($N/_{20}$)

                    CH$_3$COOH   :   ~0.05N ($N/_{20}$)

                    NaOH   :   ~0.5N ($N/_{2}$)

How to use conductivity Meter:

1. Connect the instrument to A/C mains and Switch it on.

2. Take out the conductivity cell from the water beaker. Rinse it with distilled water and wipe it with tissue paper.

3. Dip the conductivity cell in the respective (test) solution.

4. Press the cell switches to **'1'.**

5. Select the conductivity range by pressing either 200mho (for strong acid) or 20mho switch (for weak & mixture of acids).

6. For calibration-Adjust the temperature control knob to 25°C.

7. Calibrate the conductivity meter by pushing the MEAS/CAL switch to CAL position **(release the button)** and adjust CAL control knob to get 100.0 displayed on the read out (for conductivity range 200 ms range) and 10.00 is displayed (for conductivity range 20 ms)

8. For measurement - adjust the temperature control knob to the room temperature (30°C)

9. Read the conductivity of the solution by pressing CAL/MEAS switch to MEAS position **(pressed).**

10. At the end of the experiment follow the winding up instructions.

Note: Do not take out the conductivity cell from the solution when switch is in MEAS position (pressed).

B. Estimation of amount of HCl/CH₃COOH present in the given test solution using conductometry.

Procedure:

Make up the given test solution (HCl/CH₃COOH or HCl + CH₃COOH) upto the mark. Shake it thoroughly to make the solutions homogenous. 40 ml of the test solution is placed in a 100 ml beaker. The conductivity cell is dipped in this solution. The initial conductance is measured before titration. Now, NaOH is added from the burette in intervals of 0.5 ml. After the addition of each instalment of NaOH, the solution is mixed gently with a stirrer. Conductance is measured. Conductance is plotted against the volume of the alkali added. From the graph the neutralization point is obtained.

The above experiment is also done using an acid mixture (containing HCl and CH₃COOH)
From the graph equivalence points for each acid is obtained.

Application of conductometric measurements of aqueous and non aqueous solutions:

Used in cooling towers, reverse osmosis, steam boilers, acid, salt and alkali concentrations,

Industries which use conductance:-

Chemical, Power generation, hospitals, textiles, agriculture, food processing, brewing, petroleum etc.

Questions:

1. In the titration of HCl vs NaOH even at the neutralization point some conductivity is observed. Why?
2. What are the advantages of instrumental Chemical analysis over conventional volumetric analysis?
3. The initial conductance in a solution of HCl is due to which ions?
4. Name the factors which affect the conductivity of a solution?
5. How can you calculate molecular and equivalent conductivities of a solution from observed conductivities?
6. Why the concentration of base is kept 10 times higher than the concentration of acids?
7. Is it possible to determine the volume of strong and weak acids in a mixture?

Expt (1) Conductometric Titration of Strong Acid Vs Strong Base

Date:

Expt No:

TABULATION AND CALCULATIONS

Part A : Preparation of Std. Oxalic Acid Solution :

Table.1.weight of bottle + Oxalic Acid

	.			

g.

Table.2.Weight of empty weighing bottle

	.			

g.

Wt. of Oxalic Acid = W_1-W_2 =

$N_{(std)}$ Oxalic Acid = $\frac{wt}{eq.wt}$ x $\frac{1000}{Vml}$ =

(V_{ml} = 100ml)

Part B : Standardization NaOH

Titration of Std. Oxalic Acid Vs. NaOH (link)

Sl No.	$V_{oxalic\ acid}$ (ml)	Burette Reading		V_{NaOH} ml
		Initial	Final	

$N_{NaOH} = \dfrac{N_{oxalic} \times V_{oxalic}}{V_{NaOH}}$

Part – C Estimation of HCl in the test solution :

Table : Conductometric titration of HCl X NaOH

S.No.	V_{NaOH} (ml)	Conductivity (mscm^{-1})
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		
16		
17		

$$N_{HCl} = \frac{N_{NaOH} \times V_{NaOH}}{V_{HCl}}$$

=

V_{NaOH} = Inflection point from the graph

$$Wt_{HCl} = N_{HCl} \times 36.5 =$$

Results:

1. Weight of Oxalic Acid = _____ g

2. $N_{std.}$ Oxalic Acid = _____

3. N_{NaOH} (Link) = _____

4. VNaOH end point (from graph) = _____ (ml)

5. N_{HCl} (Test) = _____

6. Weight of H Cl in test solution = _____ gpl

Experiment (2): Conductometric Titration of Weak Acid Vs Strong Base

Part – C Estimation of CH₃COOH in the test solution :

Table : Conductometric titration of CH₃COOH X NaOH

S.No.	V_{NaOH} (ml)	Conductivity $(mscm^{-1})$
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		
16		
17		
18		
19		
20		

$$N_{CH3COOH} = \frac{N_{NaOH} \times V_{NaOH}}{V_{CH3\ COOH}}$$

=

V_{NaOH} = Inflection point from the graph

$Wt_{CH3\ COOH} = N_{CH3\ COOH} \times 60$ = _____ gm/lit

Marks
Observations and calculations (20):_____
Results (10):_____
Discussion of results (5):_____
Record (15):_____
Total(50):_____

Results:

1. Weight of Oxalic Acid = _____ g

2. $N_{std.}$ Oxalic Acid = _____

3. N_{NaOH} (Link) = _____

4. V_{NaOH} @ end point (from graph) = _____ (ml)

5. $N_{CH3\ COOH}$ (Test) = _____

6. Weight of CH_3 COOH test solution = _____ gpl

Signature of Faculty

*Inaction saps the vigor of the mind.

Expt (3) Conductometric Titration of Mixture of Acid Vs Strong Base

Date:

Expt No.

TABULATION AND CALCULATIONS

Part A : Preparation of Std. Oxalic Acid Solution :

Table.1.weight of bottle + Oxalic Acid

	.			

g.

Table.2.Weight of empty weighing bottle

	.			

g.

Wt. of Oxalic Acid = W_1-W_2 =

$N_{(std)}$ Oxalic Acid = $\frac{wt}{eq.wt}$ x $\frac{1000}{Vml}$ =

(V_{ml} = 100ml)

Part B : Standardization NaOH

Titration of Std. Oxalic. Vs. NaOH (link)

Sl No.	V_{oxalic} (Std) (ml)	Burette Reading		V_{NaOH} ml
		Initial	Final	

$$N_{NaOH} = \frac{N_{oxalic} \text{ X } V_{oxalic}}{V_{NaOH}}$$

Part – C Estimation of HCl and CH₃COOH in the test solution :

Table : Conductometric titration of HCl + CH₃COOH X NaOH

S.No.	V_{NaOH} (ml)	Conductivity (mscm⁻¹)
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		
16		
17		
18		
19		

Note: Vol of HCl and CH₃COOH = 40ml

$$N_{HCl} = \frac{N_{NaOH} \times V_{NaOH}}{V_{HCl}}$$

=

V_{NaOH} = Inflection point from the graph (V_1) =

$Wt_{HCl} = N_{HCl} \times 36.5$

$$N_{CH_3\,COOH} = \frac{N_{NaOH} \times V_{NaOH}}{V_{CH_3\,COOH}}$$

V_{NaOH} = Inflection point from the graph ($V_2 - V_1$)

$Wt_{CH_3\,COOH} = N_{CH_3\,COOH} \times 60 = $_____ gm/lit

Results:

1. Weight of Oxalic Acid = _____ g

2. $N_{std\text{-}Oxalic\,Acid}$ = _____

3. N_{NaOH} (Link) = _____

4. V_{NaOH} @ end point (from graph) for HCl = _____ (ml)

5. N_{HCl} (Test) = _____

6. **Weight of HCl in test solution** = _____ gpl

7. V_{NaOH} @ end point (from graph) for CH₃COOH = _____

8. $N_{CH3COOH}$ (Test) = _____

9. **Weight of CH₃COOH in test solution**. = _____ gpl

EXPERIMENT (4) <u>CONDUCTOMETRIC PRECIPITATION TITRATION</u>

AIM: To determine the amount of $BaCl_2$ present in the given solution by Conductometric titration.

Theory:- Solution of electrolytes conducts electricity due to the presence of ions. Since specific conductance of a solution is proportional to the concentrations of ions in it, conductance of solution is measured during titration.

In the precipitation titration, the ions are converted to insoluble precipitate, which will not contribute in the conductance. When Na_2SO_4 is added slowly from the burette to the solution of $BaCl_2$, Barium ions are substituted by relatively slow moving Na^+ ions. This decreases the conductance gradually and Barium sulphate ($BaSO_4$) gets precipitated while chloride ions are liberated, as shown in the equation.

$$[Ba^{+2} + 2Cl^-] + [2Na^+ + SO^{-2}_4] \rightarrow BaSO_4 \downarrow + 2Na^+ + 2Cl^-$$
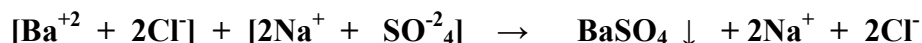
After the end-point, when all the Ba^{+2} ions are replaced, further addition of Na2SO4 increases the conductance. This is due to presence of excess of Na^+ and SO^{-2}_4 ions in the solution.

Eq wts:- $BaCl_2$ = 208.23/2=104.1 Na_2SO_4 = 142.04/2=71.02.

PROCEDURE:

PART-A: PREPARATION OF STANDARD (0.2N) Na_2SO_4 SOLUTION:

Weigh accurately about 1.42g of the given amount of Na_2SO_4 into a 100ml standard flask, dissolve in small quantity of distilled water and shake for uniform concentration.

PART-B: ESTIMATION OF $BaCl_2$:

Make up the given $BaCl_2$ solution to 100ml in a standard flask using distilled water. Pipette out 40ml of the solution into a clean 100ml beaker. Immerse a conductivity cell and note down the conductance of the solution, after calibrating the conductivity meter. Fill the burette with Na_2SO_4. Add Na_2SO_4 at intervals of 0.5ml, stir the solution well and measure the conductance of the solution on every addition. The conductometric titration is continued for further 4-5 ml after getting the end point. Conductance is plotted against the volume of Na_2SO_4 added. The point of intersection gives the end point.

Graph:- V $_{Na2SO4}$ **Vs conductivity (ms)**
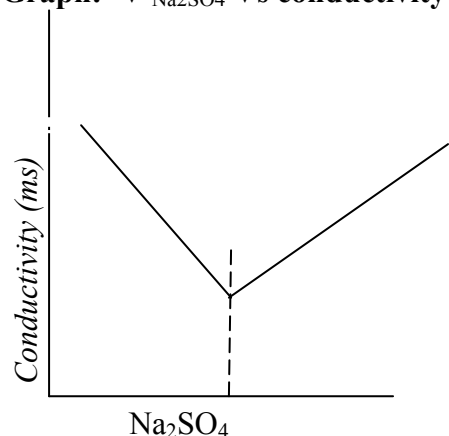
TABULATION AND CALCULATION

PART A: PREPARATION OF STANDARD (0.2N) Na₂SO₄ SOLUTION

Weight of weighing bottle + Na_2SO_4 (W_1)   = _____

Weight of empty weighing bottle (W_2)   = _____

Therefore, Weight of Salt Na_2SO_4 (W_1-W_2)   = _____

And N_{Na2SO4} =_____ =_____

PART B: ESTIMATION OF BaCl₂:

S.No.	Volume of Na₂SO₄ (ml)	Conductance in ms

Calculations:

$$N_{BaCl2} = \frac{Na_2SO_4 \times Na_2SO_4}{VBaCl_2}$$

Weight of $BaCl_2$ = $NBaCl_2$ x eq wt of $BaCl_2$ = _____g/l.

Results:

1. Weight of Na_2SO_4 =   _____g

2. N_{Na2SO4}.   =   _____

3. V_{Na2SO4} at end point
   From the graph   =   _____

4. N_{Bacl2} =   _____
5. The weight of $BaCl_2$
   Present in lit of solution = _____g/l

Marks
Observations and calculations (20):_____
Results   (10):_____
Discussion of results (5):_____
Record (15):_____
Total(50):_____

POTENTIOMETRY

THEORY:

In Potentiometry, cell potential measurement are taken for the location of the end point in titrimetric methods of analysis like acid-base, redox titrations etc

The equipment used consists of a potentiometer (a potential measuring device) a reference electrode and an indicator electrode.

The half cell potential of the reference electrode is a known constant and this electrode is completely insensitive to the composition of the solution under study. Usually, saturated calomel electrode is used as a reference electrode in potentiometric titrations.

Along with the reference electrode, an indicator or working electrode is also employed which responds to the changes in the concentration of the solution under study.

A cell is constructed by combining the reference electrode an indicator electrode and the test solution in the cell is titrated against the standard solution. During the course of the titration, the changing values of cell potential are noted down and later plotted as a graph from which end point of the titration is noted and concentration of the test solution is calculated.

Examples of Potentiometry Redox titrations:

1) Fe^{2+} x $KMnO_4$
2) Fe^{2+} x $K_2Cr_2O_7$

Examples of Potentiometry Acid – Base titrations:

i)   **Strong Acid (HCl) x Strong Base (NaOH)**
ii)   **Weak Acid (CH3COOH) x Strong Base (NaOH)**
iii)   **Mixture of Acids (HCl + CH3COOH) x Strong base (NaOH)**

*Begin Challenging your own assumptions your assumptions are your windows on the world.

Scrub them off every once in a while, or the light won't come in.
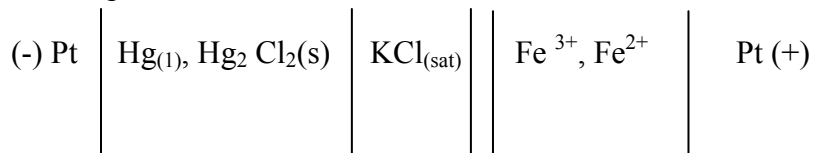
POTENTIOMETRY REDOX TITRATION

Estimation of Fe^{2+} Vs $KMnO_4$

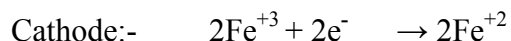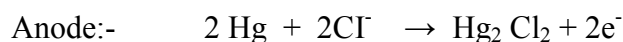**Aim:** To titrate by Potentiometry Fe^{2+} against standard **KMnO₄**

Theory : The reference electrode used here is saturated calomel electrode (SCE). It consists of mercury metal covered with a paste of $Hg + Hg_2Cl_2 \downarrow$ in contact with saturated KCl solution and Pt-Wire for electrical contact. The reduction potential of this electrode is 0.242V. This saturated calomel electrode functions as ANODE.

The **Indicator electrode** is a platinum electrode which responds rapidly to oxidation- reduction couples and senses the potential which depends upon the concentration ratio of the reactants & products of redox reactions. Here, the Pt electrode is in contact with a Ferrous-Ferric couple. This electrode functions as CATHODE.

Cell Representation:

(-) Pt | $Hg_{(1)}$, $Hg_2Cl_2(s)$ | $KCl_{(sat)}$ || Fe^{3+}, Fe^{2+} | Pt (+)

Cell Reaction:

Anode:- $2\,Hg + 2Cl^- \rightarrow Hg_2Cl_2 + 2e^-$

Cathode:- $2Fe^{+3} + 2e^- \rightarrow 2Fe^{+2}$

Cell E.m.f.:

$E_{cell} = E^o\,(Fe^{3+}/Fe^{2+}) + \dfrac{2.303RT}{F} \log \dfrac{[Fe^{3+}]}{[Fe^{2+}]} - E_{SCE}$

The cell potential is measured during the course of reaction and graphs are plotted. From the graphs end point of the titration is located and concentration is calculated.

Requirements: Saturated calomel electrode, platinum plate electrode, potentiometer, beakers (100ml) burette, pipette, stirrer and salt bridge.

Chemicals:

i)   Ferrous ammonium sulphate (~N/80) Test solution.
ii)   Potassium permanganate (~N/40) solution

PROCEDURE:

PART-A: PREPARATION OF STANDARD F.A.S. SOLUTION:

The given Mohr salt (F.A.S.) is weighed accurately in a clean weighing bottle and transferred into a clean 100 ml standard flask through a funnel. It is then dissolved in ½ test tube (~10 ml) of dil H2SO$_4$ and the solution is made up to the mark with distilled water. The solution is shaken thoroughly to make it homogeneous. From the weight of FAS the Normality of Standard solution is calculated.

PART-B: Standardization of $KMnO_4$ solution:

20ml of the prepared standard FAS solution is pippeted out into a clean conical flask. Approximately (1/2 Test Tube) 10 ml of dilute H_2SO_4(6N) is added to provide acidic medium.   It is then titrated with $KMnO_4$ taken in burette very slowly until the solution acquires pale pink colour which persists for at least a minute as end point. The titre value is noted.

The process is repeated till concordant titre values are obtained. From the titration data Normality of $KMnO_4$ is calculated.

PART C : Estimation of Fe^{+2} in the given test solution:

1. Make the given test solution of Fe^{+2}, up to the mark by adding distilled water. Make it homogeneous.

2. Connect the potentiometer to A/C mains, Switch it on.

3. Pipette out 20 ml of test solution in a clean 100 ml beaker, place the platinum electrode in the solution, which creates a Fe^{+2}/Fe^{+3} couple. Connect the electrode to pH terminal.

4. In another 100 ml beaker a Saturated calomel electrode (SCE) is placed in KCl(Sat.) solution. This is connected to R.E. terminal.

5. The two solutions are connected by means of salt bridge to form the Galvanic cell
   $$^{(-)} Pt \mid Hg_{(1)}, Hg_2\,Cl_2(s) \mid KCl_{(sat)} \mid\mid Fe^{+3}, Fe^{+2} \mid Pt^{(+)}$$

6. Switch on the instrument. Keep the mV current RED switch in pressed position (mV mode). The display reads EMF of cell in mill volts. Adjust the initial EMF to a present value with right side knob.

7. Add $KMnO_4$ from burette in 1 ml portions to the ferrous solution, stir it and note the EMF. (Table-1).
8. Continue the titration till a sudden inflextion in EMF occurs. Then take about 6 to 8 readings after inflexion in 1 ml INTERVALS.
9. From the titrations approximate volume of $KMnO_4$ required is found out.

*Invest a few moments in. It will pay good interest.

10. The titration is repeated with addition of $KMnO_4$ in 0.1 ml lots in the vicinity of end point (In 2 ml range). (Table-2).

11. Draw a graph of E_{cell} Vs volume of $KMnO_4$ added; the inflexion point gives an approximate equivalence.

12. Differential graph is drawn by plotting $\frac{\Delta E}{\Delta V}$ (Y-axis) Vs V_{KMnO4} (X-axis) to get a sharp peak, which corresponds to the precise equivalence point of titration.

TABULATION

Table : 1.

S.No.	V_{KMnO4}	Ecell
1		
2		
3		

[Draw 17 lines in full page]

Table : 2.

S.No.	V_{KMnO4} (ml)	Ecell (mv)	ΔE	$\frac{\Delta E}{\Delta V}$
1				
2				
3				

[Draw 25 lines in full page]

GRAPHS

V_{KMnO4} added (ml) (A)

V_{KMnO4} added (ml) (B)

*Genius is nothing but a great aptitude for patience..
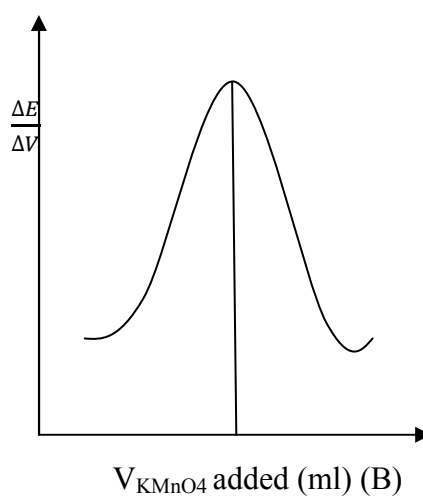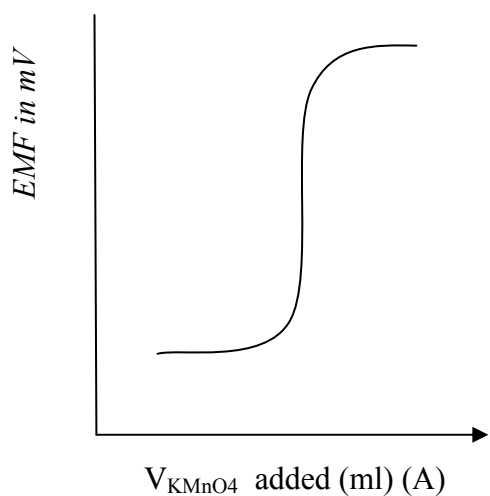
Exp. No. (5) POTENTIOMETRY REDOX TITRATION

Date:

Exp. No:

TABULATION AND CALCULATIONS

Part A : Preparation of Std. Ferrous Ammonium Sulphate Solution :

Table.1.weight of bottle + FAS

	.			

g.

Table.2.Weight of empty weighing bottle

	.			

g.

Wt. of FAS = $W_1 - W_2$ =

$N_{(std)}$ FAS $= \frac{wt}{eq.wt} \times \frac{1000}{Vml} =$

Part B : Standardization of KMnO$_4$ solution.

Titration of Std. FAS. Vs. KMnO$_4$ (link)

Sl No.	V_{Fe}^{2+} (ml)	Burette Reading		V_{KMnO4} ml
		Initial	Final	

$N_{KMnO4} = \dfrac{N_{FAS} \times V_{FAS}}{V_{KMnO4}}$

POTENTIOMETRY REDOX TITRATION (Fe^{+2} Vs. KMnO$_4$)

PART-C: Estimation of Fe^{+2} in the given test solution:

Table : 1.

S.No.	Vol of KMnO$_4$ added (ml)	E$_{cell}$ (mv)
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		
16		
17		

Table : 2.

S.No.	V$_{KMnO4}$ (ml)	E$_{cell}$ (mv)	ΔE	$\frac{\Delta E}{\Delta V}$
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				
23				
24				
25				

Calculations:

$$N_{Fe}{}^{2+} = \frac{N_{KMnO4} \times V_{KMnO4}}{V_{Fe}{}^{2+}}$$

(^{V}**KMnO$_4$** = Peak from differential graph –B)

Wt of Fe^{2+} = NFe^{2+} x 56 =   gpl =

Results:

1. Weight of FAS = _____g

2. $N_{std.}$ FAS = _____

3. N_{KMnO4} (Link) = _____

4. V_{KMnO4} @ end point (from graph) = _____ (ml)

5. NFe^{2+} (Test) = _____

6. Weight of Fe^{2+} in test solution = _____ gpl

Questions:

1. Which property or parameter is measured in Potentiometry?

2. Which electrode acts as mode and which one as cathode in this experiment?

3. What are the reactions taking place at anode and cathode?

4. What is salt bridge? What is its role?

5. Why emf of cell increases on addition of $KMnO_4$ to Fe^{2+} solution?

Marks

Observations and calculations (20):_____

Results & Graphs (10):_____

Discussion of results (5):_____

Record (15):_____

Total(50):_____

Signature of Faculty

*The world we have created is a product of our thinking;
it cannot be changed without changing our thinking.

-Einstein

EXPERIMENT-6

POTENTIOMETRY ACID-BASE TITRATION

ESTIMATION OF HCl Vs NaOH (Strong Acid Vs Strong Base)

Aim: To Titrate potentiometrically HCl against NaOH.

Theory: Reference electrode used here is saturated calomel electrode (SCE) with reduction potential 0.242V. This electrode functions as ANODE.

The indicator electrode in Quinhydrone electrode (QE). Quinhydrone is an equimolar

mixture (1:1) of Quinone (Q) and Hydroquinone (QH_2). When a pinch of quinhydrone is added to an acid solution, in contact with Pt electrode the following equilibrium is set up.

$$O=\text{⬡}=O + 2H^+ + 2e^- \rightleftharpoons HO-\text{⬡}-OH$$

     Quinone   Hydroquinone
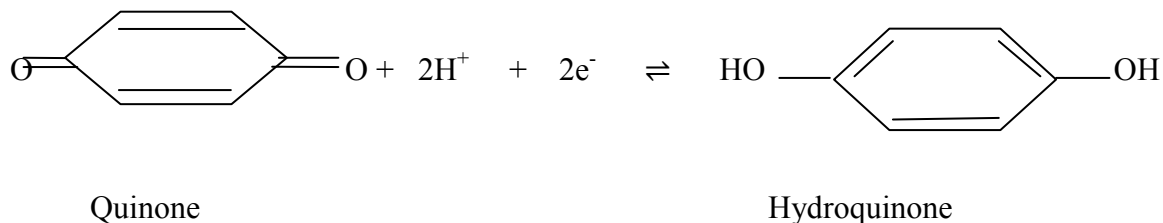
This electrode is reversible with respect to H_3O^+ ions in the solution. The reduction potential of this system is given by

$$E_{Q/QH_2} = E^o_{Q/QH_2} - 0.0591P^H$$

Where E^o Q/QH_2 (SRP of Quinhydrone electrode) is 0.6996V at 25°C. The potential of Quinhydrone electrode depends on P^H of the solution. This Quinhydrone electrode functions as CATHODE.

The electrochemical cell is constructed by combining the saturated calomel electrode (SCE) with the quinhydrone electrode (QE).

*Few minds wear out; most rust out.

Cell Representation:-

$$(-)\ Pt\ \left|\ Hg_{(1)},\ Hg_2Cl_2(s)\ \right|\ KCl_{(sat)}\ \left|\left|\ Q,\ QH_2\ \right|\ Pt^{(+)}\right.$$

Cell Reaction:-

Anode:- $\qquad\qquad 2Hg\ +\ 2Cl^-\qquad \rightarrow\ Hg_2Cl_2 + 2e^-$

Cathode:- $\qquad\qquad Q + 2H^+\ + 2e^- \rightarrow\ QH_2$
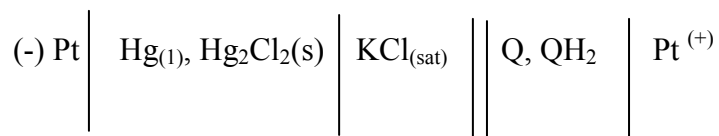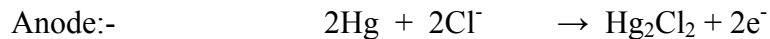
Cell E.m.f:-

$$E_{cell}\ =\quad E_{QE}\ -\ E_{SCE}$$

$$=\ \left[E_{QE}^o - 0.0591pH\right] - 0.242$$

$$=\ E_{cell}\ =\ 0.6996 - 0.0591pH - 0.242$$

$$=\ E_{cell}\ =\ 0.458 - 0.0591pH$$

$$=\ E_{cell}\ \text{is a function of pH.}$$

During the titration, as base is added to the acid, the H^+ ion concentration in the half cell containing Quinhydrone will decrease. Correspondingly, there will be a decrease in the E_{QE} and E_{cell} values also. Ecell values are noted down, graph is plotted, end point is located and concentration is calculated.

Potentiometric Acid-Base Titrations: Graphs
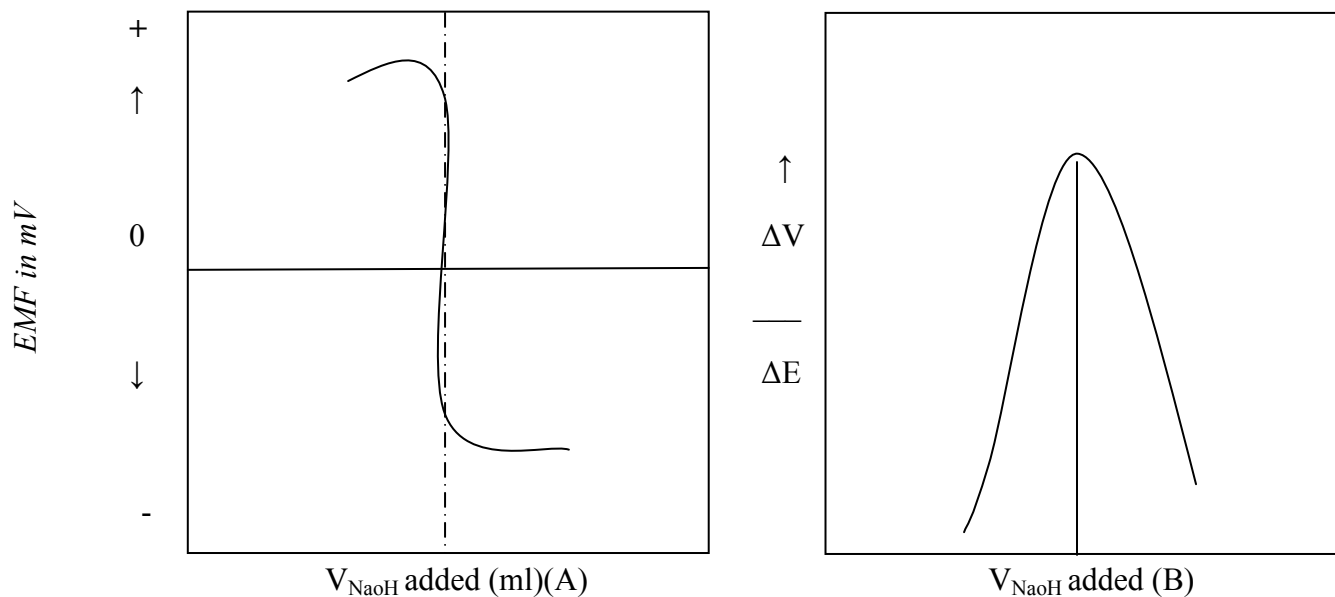
EXPERIMENT: Titration of a strong Acid (HCl) Vs strong base (NaOH)

Requirements: Saturated calomel electrode, platinum electrode, quinhydrone, potentiometer, beakers, burette, pipette, stirrer, salt bridge. etc.

Chemicals: the following solutions are required:

1) 0.1 N NaOH (N/10)
2) 0.05 N HCl (N/10)
3) Quinhydrone.

Procedure:

Part A: Preparation of standard Oxalic acid solution

Weigh about 0.63 g of oxalic acid, in a weighing bottle. Transfer it into a clean 100 ml std. flask through funnel. Dissolve it in distilled water and make a homogeneous solution upto the mark. From the wt. of Oxalic acid calculate the normality of std. solution.

Part B: Standardization of NaOH solution.

Take NaOH(~N/10) solution in burette. Pippete 20ml of standard Oxalic acid solution in a clean conical flask. Titrate it with NaOH using phenolphthalein indicator till colourless solution changes to pale pink in colour. From the titration data normality of NaOH solution is calculated.

Part C: Estimation of HCl in the given test solution

1. Make the given test HCl solution homogeneous by adding distilled water upto the mark.

2. Connect the Potentiometer to A/C mains and Switch it on.

3. Pipette 20ml of the test soln. in a clean 100 ml beaker. Add a pinch of Quinhydrone to it and dip a platinum electrode in the solution. Connect the electrode to pH terminals.

*He who will not reason is a bigot; he who cannot is a fool; and he who dares not is a sane.

4. In another 100 ml beaker, a saturated calomel electrode is placed in a solution of KCl(Sat.). The electrode is connected to RE terminals.

5. The two solutions are connected by means of salt bridge to form a Galvanic cell.
$$^{(-)} Pt \mid Hg_{(1)}, Hg_2Cl_2(s) \mid KCl_{(sat)} \parallel H^+ \text{ (unknown)} \mid Q, QH_2 \mid Pt^{(+)}$$

6. Switch on the Instrument; keep the mv/current RED switch in pressed position (mv mode). Display reads the EMF of cell in milli volts. Adjust the initial EMF to a present value.

7. Add NaOH from burette in 1 ml portions to the acid, stir it and note the EMF.

8. **Continue the titration till a sudden inflexion in EMF occurs (the value changes to –ve), then take about 6 to 8 readings after inflexion in 1 ml intervals.**

9. From the titration, approximate volume of NaOH required is found out. (Table-1)

10. The titration is repeated with addition of NaOH in 0.1 ml lots in the vicinity of end point (In 2 ml range) (Table -2)

11. Draw a graph between E cell Vs volume of NaOH. Added. The intersection point at Ecell = Zero may give an approximate end point.

12. Another graph is obtained by plotting $\Delta E/\Delta V$ (y-axis) vs V_{NaOH} (x-axis) to get a sharp peak, which corresponds to the equivalence point.

Questions:

1. What is the relation between emf of cell and pH of solution?

2. Why cell potential decreases with addition of alkali to the test acid solution?

3. What is the use of quinhydrone in this experiment?

Expt (6) POTENTIOMETRY ACID-BASE TITRATION

Date:

Expt No.

TABULATION AND CALCULATIONS

Part A : Preparation of Std. Oxalic Acid Solution :

Table.1.weight of bottle + Oxalic Acid

	.			

g.

Table.2.Weight of empty weighing bottle

	.			

g.

Wt. of Oxalic Acid = $W_1 - W_2$ =

$N_{(std)}$ Oxalic Acid $= \dfrac{wt}{eq.wt} \times \dfrac{1000}{Vml} =$

(V_{ml} = 100ml)

Part B : Standardization of NaOH

Titration of Std. Oxalic. Vs. NaOH (link)

Sl No.	V_{oxalic} (Std) (ml)	Burette Reading		V_{NaOH} ml
		Initial	Final	

$$N_{NaOH} = \frac{N_{oxalic} \times V_{oxalic}}{V_{NaOH}}$$

POTENTIOMETRY ACID-BASE TITRATION (HCl Vs. NaOH)

PART-C: Estimation of HCl in the given test solution:

Table : 1.   Table : 2.

S.No.	Vol of NaOH added (ml)	Ecell (mv)
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		
16		
17		

S.No.	V_{NaOH} (ml)	Ecell (mv)	ΔE	$\dfrac{\Delta E}{\Delta V}$
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
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24				
25				

Calculations:

$$N_{HCl} = \frac{N_{NaOH} \times V_{NaOH}}{V_{HCl}}$$

(^{V}NaOH = Peak from differential graph – (B)

Wt of HCl   = N_{HCl} x 36.5 = _____gpl

Results:

1. Weight of Oxalic Acid   =   _____g

2. $N_{std.}$ Oxalic Acid   =   _____

3. N_{NaOH} (Link)   =   _____

4. V_{NaOH} @ end point (from graph)   =   _____ (ml)

5. N_{HCl} (Test)   =   _____

6. Weight of HCl in test solution   =   _____ gpl

Marks

Observations and calculations (20):_____

Results & Graphs   (10):_____

Discussion of results (5):_____

Record (15):_____

Total(50):_____

Signature of Faculty

*A lot of what passé for depression these days is nothing more than the body saying that it needs work.

g e

pH Metry

Glass Electrode ---- pH

THEORY:- Glass electrode is a pH indicating electrode, which is H^+ ion sensitive.

Construction:

A special type of soft-glass of the soda-lime type, with a low melting point, relatively high electrical conductivity, is blown into a thin walled bulb. This bulb is fused to an ordinary glass-tube. Aqueous solutions of 0.1N HCl and saturated KCl are sealed within the bulb. A platinum wire coated with Ag-AgCl, immersed in this solution, is an internal reference electrode that provides the electrical contact.

When the glass electrode, containing 0. 1N HCl is placed in a solution of a different pH, will develop a difference in potential at the interface between the glass membrane and the solutions containing the H^+ ions. The magnitude of this difference of potential depends upon the difference in the concentrations of H^+ ions in both the solutions.

Cell Notation:-

(-) Pt	$Hg_{(1)}$, $Hg_2\ Cl_2(s)$	Sat. KCl	Test soln of Unknown pH	Glass membrane	0.1N HCl	AgCl, Ag	$Pt^{(+)}$

Cell Reaction :-

Anode:- $2Hg\ +\ 2Cl^-\ \rightarrow\ Hg_2\ Cl_2 + 2e^-$

Cathode:- $2AgCl + 2e^-\ \rightarrow\ 2Ag^+ + 2Cl^-$
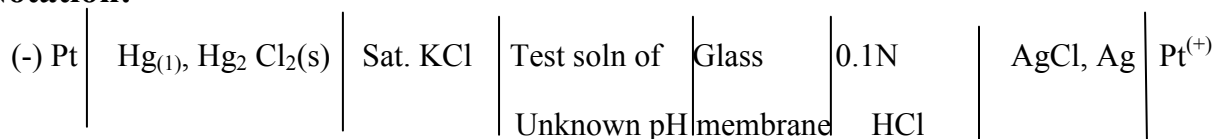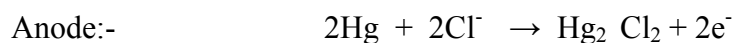
The EMF of cell is given as

$$E_{cell} =\quad E_{Glass}\ -\ E_{SCE}$$

The potential of Glass electrode is given by

$$E_{Glass}\quad =\quad [E_G^o - 0.0591pH] \qquad E_G^o = SRP\ of\ Glass\ electrode$$

$$=\ E_{cell} = E_G^o - 0.0591pH - 0.242$$

$$pH = \frac{E_G^{\ o}\ -\ E_{cell} - 0.242}{0.0591}$$

$$(P^H = ^-log[H^+])$$

pH Meter: The glass membrane of the glass electrode offers a high resistance in measuring circuit of the potentiometer and even a highly sensitive galvanometer fails to detect currents flowing due to potential imbalance. For null point detection, and for measuring the small current, amplification of signals is necessary. pH meter is the instrument used for this purpose. It is an electronic voltmeter of requisite sensitivity and stability, and it provides a scale calibrated directly in pH units.

P^H Scale = Acidic = 1 to 7, Basic = 7 to 14

ACID –BASE pH-METRIC TITRATION

THEORY:

1) Titration of strong acid (HCl) against a strong base (NaOH):

In this titration, as the base is added to the acid, the OH^- ions of the base react with the H_3O^+ ions of the acid forming water.

$$H_3O^+ + OH^- \rightleftharpoons 2H_2O$$

Consequently, the H_3O^+ ion concentration in the acid will decrease, i.e., the pH of the acid will progressively increase. Initially the change in the pH will be gradual, however near the neutralization point we will have a sharp increase in the pH from about 4 to 10 within 0.2 ml range of the base added, around the end point.

From the plot of the pH against the volume (V) of the base added, we will obtain the end point of the titration.

Plot of $\frac{\Delta pH}{\Delta V}$ Vs V in the vicinity of the equivalence point (V=0.1 ml) will give a curve whose peak indicates a sharp end point.

2) Titration of a weak acid (CH₃COOH) against a strong base (NaOH):

Here the initial concentration of H_3O^+ ions in acid solution will be low, hence the high pH. As the titration proceeds the pH of the acid will gradually increase. At the equivalence point, change in the pH will be sharp, as in titration of a strong acid and strong base, but the slope of the curve, obtained in the plot of pH vs Volume of the base added, is not as steep as in the strong acid strong base titration curve. Another point to be noted in this titration is that the anionic (CH₃COO⁻) hydrolysis of the salt formed, will make the pH of the reaction mixture higher than 7 at the equivalence point.

Volume of base added→(A)

V_NaOH added (ml)

(B)
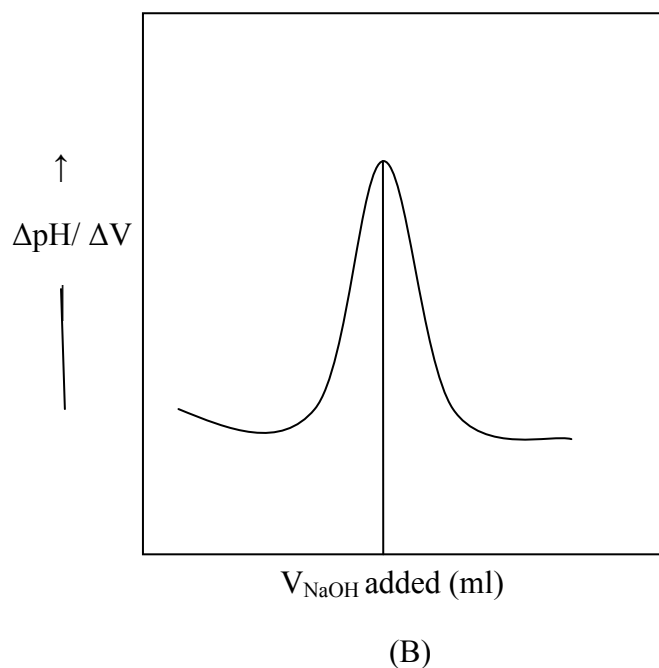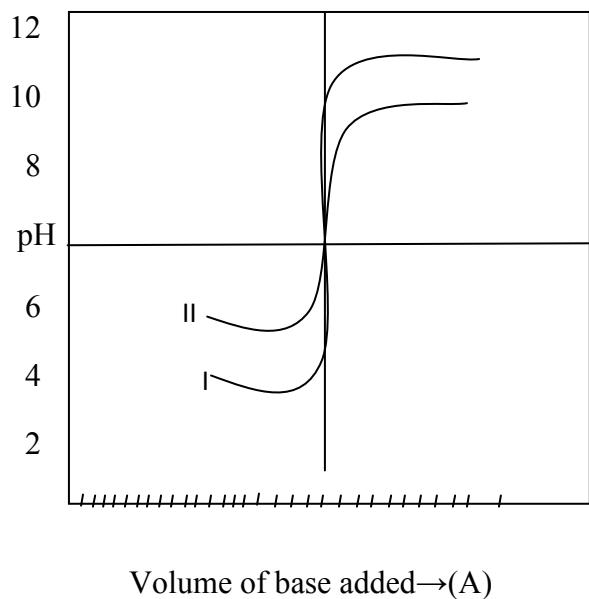
Neutralization of **I** strong acid and strong base

II Weak acid and strong base

Experiment : (7) Titration of strong acid vs strong base.

Requirements : pH meter, glass electrode, burette, pipette, beakers.

Chemicals : HCl (N/40); NaOH (N/10)

Procedure:

Part 'A': Preparation of standard Oxalic acid solution.

Weigh about _____ gms of oxalic acid in a weighing bottle   Transfer it into a clean 100 ml standard flask through funnel. Dissolve it in distilled water to make a homogeneous solution upto the mark. From the wt of oxalic acid calculate the normality of standard solution.

Part 'B': Standardization of NaOH solution.

Take NaOH 0.1N Solution in burette. Pipette out 20 ml of standard oxalic acid solution into a clean conical flask. Titrate it with NaOH using phenolphthalein indicator till colourless soln. changes to pink in colour. From titration data, normality of NaOH solution is calculated.

Part 'C': Estimation of HCl in the given test solution

1. Switch on the pH meter by placing the plug in the A.C. mains socket.
2. Keep the Temp at 30°C and place both the switches in pressed position.
3. Place the combined Glass-calomel electrode setup in distilled water and check for display of pH=7.0 otherwise adjust with pH 7.0 preset control to get 7.0 pH (right side knob).
4. Take out the electrode from water, clean it with tissue paper and dip it in a 4.0 pH buffer.
5. Release the check / Read switch and adjust the SET Buffer control to get a display of pH=4.0 (mv=176). Once calibrated, this knob should not be disturbed.
6. Take out the electrode from Buffer soln., wash the electrode with distilled water, and clean it with tissue paper.
7. **Make the given HCl test soln. homogeneous upto the mark by adding distilled water.**
8. Pipette out 40ml of HCl in a clean 100ml beaker. Dip the electrode in the solution and measures the pH of solution by releasing the Check/Read Switch.
9. Add(~N/10) Soln. of NaOH from burette into the acid, in 1 ml intervals, stir the soln. in beaker with glass-rod and measure its pH.
10. Continue the titration till a sudden inflexion in pH occurs, then take about 6 to 8 readings after inflexion in 1 ml intervals.
11. From the titration approximate volume of NaOH required is found out. (Table-1).
12. The titration is repeated with addition of NaOH in 0.1 ml lots in the vicinity of end point (In 2ml range) (Table-2).
13. Draw a graph of pH Vs. Volume of NaOH added. The intersection point pH=7.0. may give a rough end point.
14. Another graph is obtained by plotting $\Delta pH/\Delta V$(Y- axis) Vs. VNaOH (X-axis) to get a sharp peak, which corresponds to the equivalence point. (Differential Graph)

*Character is higher than intellect.

Exp. No. (7) pH- METRIC TITRATION (STRONG ACID Vs STRONG BASE)

Date:

Exp. No:

TABULATION AND CALCULATIONS

Part A : Preparation of Std. Oxalic Acid Solution :

Table.1.weight of bottle + Oxalic Acid

	.			

g.

Table.2.Weight of empty weighing bottle

	.			

g.

Wt. of FAS = W_1-W_2 = _____ = _____

$N_{(std)}$ Oxalic acid $= \dfrac{wt}{eq.wt} \times \dfrac{1000}{Vml} =$ _____ $=$ _____
(V_{ml}=100ml).

Part B : Standardisation of NaOH

Titration of Std. Oxalic Vs. NaOH (link)

Sl No.	V_{Oxalic} (Std) (ml)	Burette Reading		V_{NaOH} ml
		Initial	Final	

$N_{NaOH} = \dfrac{N_{Oxalic} \times V_{Oxalic}}{V_{NaOH}} =$ _____ $=$ _____

PART-C: Estimation of HCl in the given test solution:

Table : 1.   Table : 2.

S.No.	Vol of NaOH added (ml)	pH	Ecell (mv)
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			
15			
16			
17			

S.No.	VNaOH (ml)	pH	ΔpH	$\frac{\Delta pH}{\Delta V}$
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				
23				
24				
25				

Calculations:

$$N_{HCl} = \frac{N_{NaOH} \times V_{NaOH}}{V_{HCl}} = \underline{\hspace{3cm}} = \underline{\hspace{4cm}}$$

(V_{NaOH} = Peak from differential graph –(B)

Wt of HCl   = N_{HCl} x 36.5 = _____ = _____gpl

Application of pH measurements

- Food processing, dairies, breweries, distilleries.
- Purification of drinking water, pollution control, sewage treatment.
- Aquaculture and agricultural soil testing. Pharmaceuticals, cosmetics.
- Electroplatings, cement, fuels etc.

Questions:

1. What do you mean by combined electrode?
2. Which electrode acts as anode and which acts as cathode?
3. Why should we calibrate the instrument using a buffer solution?
4. At neutralization point what is the emf of the cell?
5. Why is salt bridge not used in this experimental set up?

Results:

1. Weight of Oxalic Acid = _____ g

2. $N_{std.}$ Oxalic Acid = _____

3. N_{NaOH} (Link) = _____

4. V_{NaOH} @ end point (from graph) = _____ (ml)

5. N_{HCl} (Test) = _____

6. Weight of $_{HCl}$ in test solution = _____ gpl

Marks

Observations and calculations (20):_____

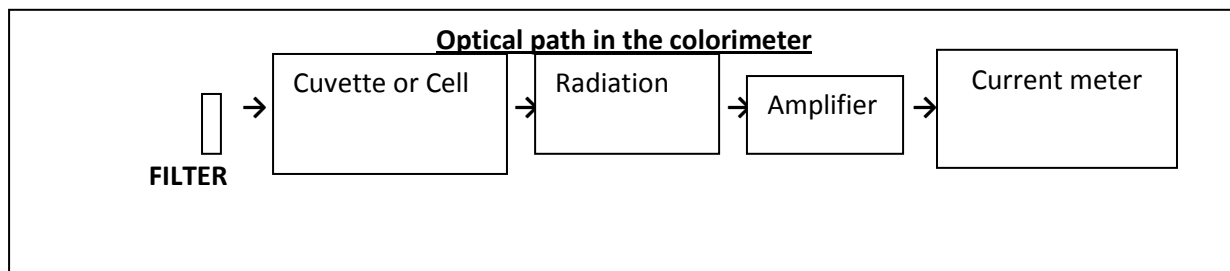Results & Graphs (10):_____

Discussion of results (5):_____

Record (15):_____

Total(50):_____

COLORIMETRY

1. **Introduction:** In colorimetry, the light absorptive capacity of a system (coloured solution) is measured and this measurement is related to the concentration of the coloured substance in the solution. The absorbance is governed by "Beer-Lambers" Law.

Optical path in the colorimeter

FILTER → Cuvette or Cell → Radiation → Amplifier → Current meter

2. **Colorimeter:**
   A colorimeter (absorptiometer) consists of the following parts:
   1) light source 2) light filter 3) cell for coloured solution 4) photocell 5) amplifier

3. **Colour Filters:**
   The filter is a coloured glass plate or coloured gelatin coated on glass plate.
   Property of a filter is that when while light is passed through the filter, it transmits light from a specified region of spectrum, preferentially absorbing the other parts of the spectrum.

4. **BEER'S LAW:** It states that when monochromatic light passes through a transparent medium the rate of decrease in intensity with the concentration of the medium is directly proportional to the intensity of the light. This is equivalent to stating that the intensity of the transmitted light decreases exponentially as the concentration of the absorbing medium increases arithmetically. It can be expressed as

   $$\frac{-dI}{dc} = KI,$$ on Integration between limits Io—It & C=0—c, gives

   $$\frac{Io}{It} = Kc \qquad \text{(or)} \qquad I_t = I_o.10^{-kc} \ (1)$$

   Where $I_0{}^o$ = Intensity of the incident light,
   I_t = Intensity of transmitted light,
   c= Concentration of the medium and
   K= proportionality factor.
   k= constant for the wave length & the absorbing medium
   $$K = \frac{k}{2.303}$$

5.LAMBERT'S LAW:- It states that when monochromatic light passes through a transparent medium the rate of decrease in intensity with the thickness of the medium is directly proportional to the intensity of the light (or) The intensity of the transmitted light decreases exponentially as the thickness of the absorbing medium increases arithmetically.

It can be expressed as $\frac{-dI}{dc} = KI$

On integration between limits Io----It and dx-0-x, we get (Where 'x' is thickness of the medium.)

$$I n \frac{I_o}{I_t} = Kx \qquad (OR) \qquad I_t = I_o . 10^{-kx} \qquad - \qquad (2)$$

6. BEER-LAMBERT'S LAW:- It is a combination of the above two laws relating intensity of light with concentration as well as thickness of absorbing medium. It states that

" when monochromatic light passes through a transparent medium the rate of decrease in intensity with the **concentration and thickness of the medium** is directly proportional to the intensity of the light."

Combination of equations 1 & 2 is Beer-Lamberts' law, which can be expressed as

$$I_t = I_o . 10^{-\epsilon cx} \qquad (OR) \quad \log \frac{I_o}{I_t} = \epsilon cx \qquad - \qquad (3)$$

Where ϵ is molar absorption coefficient or molar absorptivity, or molar extinction coefficient 'c' is expressed in moles / litre, and x in centimeters.

Equation (3) is the fundamental equation of colorimetry & spectrophotometry, and is often termed as the Beer-Lambert's Law.

The relation between absorbance A, the transmittance T and the molar absorption coefficient (ϵ) is given

$$A = \epsilon cx = \log \frac{I_o}{I_t}$$

We know that $A = \log \frac{I}{T} = -\log T$

$$\therefore \log T = \log \frac{I_t}{I_o} \qquad - \qquad (4)$$

Transmittance, T is given as $T = \frac{I_t}{I_o}$

$\%T = 100 \times I_t/I_o$

7. CONDITIONS FOR BEER-LAMBERT'S LAW TO BE APPLICABLE:.

1) Solution should be coloured.

2) Incident radiation should be monochromatic.

3) Solution should be homogeneous

4) Solution should be dilute.

5) Each molecular or ion species should absorb independently.

<u>**Principle of absorbance of radiation**</u>

Dx   Soln. of Conc. C mol.1^{-1}
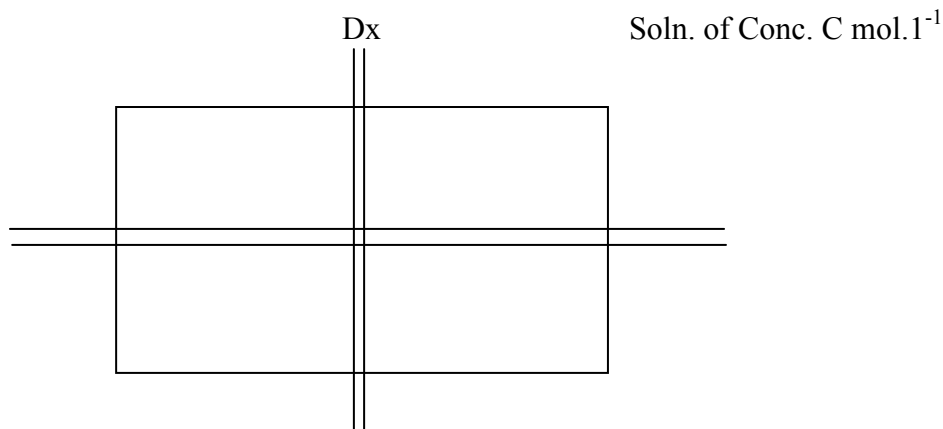
8.Principle of Operation of the Colorimeter:

A low voltage lamp forms the light source. This light passes through a select filter. The light transmitted by the filter passes through the cell containing the coloured solution, and falls on a sensitive photocell, An amplifier amplifies the current generated by the photocell. The amplifier output drives a current meter calibrated in optical density and % transmittance.

Complementary Colours for Colorimetry

Wavelength of light Transmitted by the filter (nm)	Colour (Absorbed)	Complementary Colour (Transmitted)
400-435	Violet	Yellowish green
435-480	Blue	Yellow
480-490	Greenish Blue	Orange
490-500	Bluish Green	Red
500-560	Green	Purple
560-580	Yellowish green	Violet
580-595	Yellow	Blue
595-610	Orange	Greenish Blue
610-750	Red	Bluish Green

9. Operation of the Colorimeter:

(Note: Before operation of the colorimeter, make sure that all switches are in 'off' position.

i)   Connect the colorimeter to the A/C Mains and Switch it on.

ii) 'On' the switch located at the back panel of the colorimeter. (The 'read out' will show 1.00)

iii) By rotating the disc 'B' (see diagram on Front Panel of the colorimeter), any colour filter that gives the desired wavelength range of transmitted light, can be brought in the path of the light. Select a colour filter which gives maximum optical density for a solution of a given solution under measurement.

(For the expt. With $KMnO_4$, the wavelength of transmitted light used is 490 nm or 4900 A^o (colour filter 52).

Place the solution cell with blank (distilled water is the blank for this expt.) in the housing 'C'.

(Note: The solution cell should be filled to a height not exceeding ¾ and outside dried with a tissue paper)

Adjust the disc A for zero O.D, for distilled water (blank).

iv) Replace Blank in the solution cell with various sample solutions prepared(test tube-1 to 10), one after the other (as indicated in the table), after rinsing with the respective solution.

v) Record the O.D., for each of the sample solutions.
(The O.D. value should be between 0.3. and 0.7 or else the sample solutions required for any species should be brought in this range by suitable dilution.)

The graph between the optical density, taken on the y-axis and concentration on the x-axis will give a straight line passing through the origin, with a slope of Ix. (select a suitable scale on the graph paper).

EXPT (8) COLORIMETRY: VERIFICATION OF BEER-LAMBERT'S LAW & ESTIMATION OF Mn in $KMnO_4$

PROCEDURE:

PART A: PREPARATION OF STD. FERROUS AMMONIUM SULPHATE SOLUTION:

Weigh accurately about 0.98gm of Mohr's salt and transfer it into a clean 100 mil flask using funnel. Dissolve it in distilled water, add-10 ml dil H_2SO_4 and make the solution homogeneous upto the mark.

PART B: STANDARDIZATION OF KMnO₄ USING MOHR'S SALT:

Fill the burette with KMnO₄ soln. Pipetteout 20 ml of Mohr salt soln. in a clean conical flask. Add ¾ test tube of dil H₂SO₄ and titrate the contents of flask with KMnO₄ till a light pink colour appears. From titration data, calculate the Normality of KMnO₄ (Link) solution.

Convert the concentration from Normality to Molarity.(this gives Molarity of (Link solution)

Let $M_{KMnO4} = N_{KMnO4} /5$

$M_{KMnO4} = $ 0.00048 M $= 4.8 \times 10^{-4}$ M. $\frac{0.024}{5}$

PART C: ESTIMATION OF KMnO₄ BY COLORIMETRY :

1)**Preparation of KMnO₄ stock solution:** 10ml of link KMnO4 is taken in standard volumetric flask and diluted to 100 ml., made upto the mark and homogenous to get stock solution .

2) **Preparation of sample solutions**: The above stock solution from volumetric flask is pipettd out in test tubes from 1 to 10 respectively and diluted by adding distilled water, to make 10 ml solution in each test tube. Dilution is done as given in Table 2.of the experiment.

3) **Selection of Filter No for KMnO4 solution:** The colorimeter is calibrated by taking blank solution. For each filter No. 45 to 67 the OD of stock solution is measured in Table 1.The filter No. which gives maximum OD is selected.(for KMnO4 the filter No.52 gives maximum OD.

4)**VERIFICATION OF BEER-LAMBERTS LAW:**

The selected filter no for KMnO₄ solution is fixed, The caliberation is done by taking(distilled water) blank and adjusting OD to zero, The cuvvete is filled with each of the ten sample solutions prepared, one after the other. After rinsing with respective solution. The OD is recorded for each of the sample solution. OD Values are noted in Table-2. OD of test solution is also noted.

5)**Calibration Graph:**A graph is plotted between optical density against concentration of KMnO₄ . it gives a straight line passing through origin. It is called calibration graph. The graph passing through origin is a proof for verification of Beer-Lambert's Law.

This graph can be used for estimation of the concentration of a given species (KMnO₄ in this case) in any Test solution.

6)**Concentration of test solution**:OD of test solution is marked on y-axis of the gragh. The concentration of test solution is obtained by intrapolation on x-axis.

Molarity of test solution is calculated and test solution is estimated .

COLORIMETRY:- Calibration Curve of Concentration of KMnO4 versus O.D. Verification of Beer-Lambert's Law

0.0 0.1 0.2 0.3 0.4 0.5 0.6 0.7 0.8 x(--)-
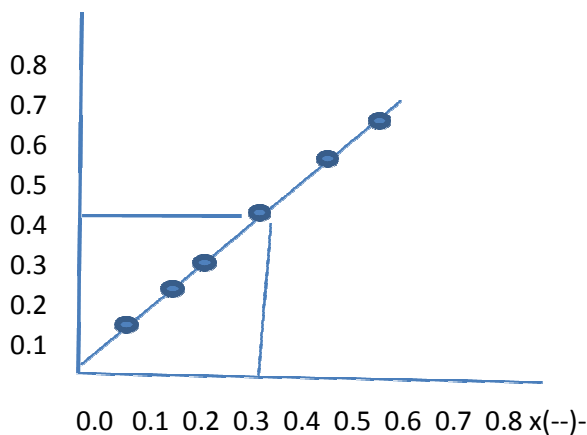
Test Solution
O.D = _____
Conc. Of $KMnO_4$ = _____ $x10^{-4}M$

Concentration of KMnO4 (_____$x10^{-4}M$)

Note the O.D. of test solution and mark it on the graph. The concentration of the test solution would be arrived at by multiplying the concentration as read on the calibration graph with the dilution factor.

Applications of Colorimetry:-

1. Estimation of steel analysis, fluoride, nitrate analysis, biochemical/laboratories, pharmaceutical industry, textile industry etc.

Questions

1. For the following transmittance what is the absorbance or optical density.

   0% ; 1% ; 10% ; 100%

2. What type of solutions are analyzed in Colorimetry?
3. How can Colorimetry be used to find out concentration of species in solutions which are not coloured?
4. Why only dilute solutions are used in Colorimetry?
5. What is the role of filters in Colorimetry and how do you select a proper filter

Exp. No. (8) COLORIMETRY: VERIFICATION OF BEER-LAMBERT'S LAW

Date:

Exp. No:

TABULATION AND CALCULATIONS

Part A : Preparation of Std. Ferrous Ammonium Sulphate Solution :

Table.1.weight of bottle + FAS

	.			

g.

Table.2.Weight of empty weighing bottle

	.			

g.

Wt. of FAS = $W_1 - W_2$ =

$N_{(std)}$ FAS = $\frac{wt}{eq.wt}$ x $\frac{1000}{Vml}$ =

(V_{ml} = 100ml)

Part B : Standardization of KMnO$_4$ solution.

Titration of Std. FAS. Vs. KMnO$_4$ (link)

Sl No.	$V_{Fe^{2+}}$ (ml)	Burette Reading		V_{KMnO4} ml
		Initial	Final	

Marks

Observations and calculations (20):_____

Results & Graphs   (10):_____

Discussion of results (5):_____

Record (15):_____

Total(50):_____

$N_{KMnO4} = \dfrac{N_{FAS} \times V_{FAS}}{V_{KMnO4}}$

Table : 1. :Selection of filters

Table : 2.

S.No.	Filter No	OD.
1	45	
2	47	
3	51	
4	52	
5	54	
6	57	
7	60	
8	67	

S.No.	Std. Soln V(KMnO4 +distilled water) (in ml)	Concentration (Molar)]	Optical Density
1	1+9	$0.1 \text{ x---x} 10^{-4}$	
2	2+8	$0.2 \text{ x---x} 10^{-4}$	
3	3+7	$0.3 \text{ x---x} 10^{-4}$	
4	4+6	$0.4 \text{ x---x} 10^{-4}$	
5	5+5	$0.5 \text{ x---x} 10^{-4}$	
6	6+4	$0.6 \text{ x---x} 10^{-4}$	
7	7+3	$0.7 \text{ x---x} 10^{-4}$	
8	8+2	$0.8 \text{ x---x} 10^{-4}$	
9	9+1	$0.9 \text{ x---x} 10^{-4}$	
10	10+0	$1.0 \text{ x---x} 10^{-4}$	
11	Test Solutions		

CALCULATIONS:

$N_{KMnO4 \text{ (stock)}}$ $= N_{(Link)}$ $\quad x \quad \dfrac{10}{100} =$

$N_{KMnO4 \text{ (stock)}}$ $= \dfrac{N(Stock)}{5} =$

O.D. of test soln =   M solution (Test) =

(From graph)

Wt. of $KMnO_4$ = $M_{(test)}$ x 158 =

=…………………..gpl

Results:

1. Weight of FAS   =   _____g

2. $N_{std.}$ FAS   =   _____

3. N_{KMnO4} (Link)   =   _____

4. N_{KMnO4} =   =   _____ (stock solution)

5. Beer-Lambert's Law verified

6. Test Solution : OD=_____: Conc of $KMnO_4$ = _____M

7. Wt of KMnO4 in test solution = _____gpl.

DEMO EXPERIMENT OF Conductometric Titration

Part – C Estimation of HCl in the test solution:

S.No.	V_{NaOH} (ml)	Conductivity (mscm^{-1})
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		
16		
17		
18		
19		

$$N_{HCl} = \frac{N_{NaOH} \times V_{NaOH}}{V_{HCl}}$$

$$=$$

V_{NaOH} = Inflection point from the graph (V_1) =

Wt_{HCl} = N_{HCl} x 36.5

Results:

1. Weight of Oxalic Acid   =   _____ g

2. N $_{std\ Oxalic\ Acid}$ =   _____

3. N_{NaOH} (Link)   =   _____

4. V_{NaOH} end point (from graph)   =   _____ (ml)

5. N_{HCl} (Test)   =   _____

6. Weight of HCl in test solution   =   _____ gpl

DEMO OF POTENTIOMETRIC TITRATION

Date :

POTENTIOMETRY REDOX TITRATION (Fe^{+2} Vs. K$_2$Cr$_2$O$_7$)

PART-C: Estimation of Fe+2 in the given test solution

Table : 1.

S.No.	Vol of K$_2$Cr$_2$O$_7$ added (ml)	Ecell (mv)
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		
16		
17		

Table : 2.

S.No.	V K$_2$Cr$_2$O$_7$ (ml)	Ecell (mv)	ΔE	$\dfrac{\Delta E}{\Delta V}$
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				
23				
24				
25				

Calculations:

$$N_{Fe}^{2+} = \frac{N_{K2Cr2O7} \times V_{K2Cr2O7}}{V_{Fe}^{2+}}$$

($V_{K2Cr2O7}$ = Peak from differential graph –B)

Wt of $_{Fe}^{2+}$ = N_{Fe}^{2+} x 56 = _____gpl

DEMO EXPERIMENT OF pH – METRIC TITRATION

Date :

PART-C: Estimation of HCl in the given test solution

Table : 1.   Table : 2.

S.No.	Vol of NaOH added (ml)	pH	Ecell (mv)
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			
15			
16			
17			

S.No.	V NaOH (ml)	pH	ΔpH	$\frac{\Delta pH}{\Delta V}$
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				
23				
24				
25				

Calculations:

$$N_{HCl} = \frac{N_{NaOH} \times V_{NaOH}}{V_{HCl}}$$

(V_{NaOH} = Peak from differential graph –B)

Wt of HCl   = N_{HCl} x 36.5 = _____ gpl

DEMO EXPERIMENT COLORIMETRY : VERIFICATION OF BEER-LAMBERT'S LAW

Table : 1. :Selection of filters   Table : 2.

S.No.	Filter No	OD.
1	45	
2	47	
3	51	
4	52	
5	54	
6	57	
7	60	
8	67	

S.No.	Std. Soln V(KMnO4 +distilled water) (in ml)	Concentration (Molar)]	Optical Density
1	1+9	$0.1 \text{ x---x}10^{-4}$	
2	2+8	$0.2 \text{ x---x}10^{-4}$	
3	3+7	$0.3 \text{ x---x}10^{-4}$	
4	4+6	$0.4 \text{ x---x}10^{-4}$	
5	5+5	$0.5 \text{ x---x}10^{-4}$	
6	6+4	$0.6 \text{ x---x}10^{-4}$	
7	7+3	$0.7 \text{ x---x}10^{-4}$	
8	8+2	$0.8 \text{ x---x}10^{-4}$	
9	9+1	$0.9 \text{ x---x}10^{-4}$	
10	10+0	$1.0 \text{ x---x}10^{-4}$	
11	Test Solutions		

CALCULATIONS:

$N_{KMnO4 \text{ (stock)}}$ $= N_{(Link)}$ x $\dfrac{10}{100} =$

$N_{KMnO4 \text{ (stock)}}$ $=$ $\dfrac{N(Stock)}{5} =$

O.D. of test soln =   M solution (Test) =

                              (From graph)

Wt. of $KMnO_4$ = $M_{(test)}$ x 158 =   =………………….gpl

Results:

1. Weight of FAS   =   _____g

2. $N_{std.}$ FAS   =   _____

3. N_{KMnO4} (Link)   =   _____

4. N_{KMnO4} =   =   _____ (stock solution)

5. Beer-Lambert's Law verified

6. Test Solution : OD=_____ :   Conc of $KMnO_4$ = _____M

7. Wt of KMnO4 in test solution =   _____gpl.

MUFFAKHAM JAH COLLEGE OF ENGINEERING & TECHNOLOGY

B.E. I YEAR II-SEMESTER INSTRUMENTAL CHEMICAL ANALYSIS

SCHEDULE OF PRACTICALS

(w.e.f. 2017-2018 Academic Year)

LAB I					LAB II			
Seat No.	I st WEEK	II nd WEEK	III rd WEEK		Seat No.	I st WEEK	II nd WEEK	III rd WEEK
1,2	C_1	C_1	Col_1		1,2	P_1	P_1	PH_1
3,4	C_2	C_2	Col_2		3,4	P_2	P_2	PH_2
5,6	C_3	C_3	Col_3		5,6	P_3	P_3	PH_3
7,8	C_4	C_4	Col_4		7,8	P_4	P_4	PH_4
9,10	C_5	C_5	Col_5		9,10	P_5	P_5	PH_5
11,12	C_6	C_6	Col_6		11,12	P_6	P_6	PH_6
13, 14	C_7	C_7	Col_7		13, 14	P_7	P_7	PH_7
15,16	C_8	C_8	Col_8		15,16	P_8	P_8	PH_8
17,18	C_9	C_9	Col_9		17,18	P_9	P_9	PH_9
19,20	C_{10}	C_{10}	Col_{10}		19,20	P_{10}	P_{10}	PH_{10}
21,22	C_{11}	C_{11}	Col_{11}		21,22	P_{11}	P_{11}	PH_{11}
23,24	C_{12}	C_{12}	Col_{12}		23,24	P_{12}	P_{12}	PH_{12}
25,26	C_{13}	C_{13}	Col_{13}		25,26	P_{13}	P_{13}	PH_{13}
27,28	C_{14}	C_{14}	Col_{14}		27,28	P_{14}	P_{14}	PH_{14}
29,30	C_{15}	C_{15}	Col_{15}		29,30	P_{15}	P_{15}	PH_{15}
31P,32	C_{16}	C_{16}	Col_{16}		31P,32	P_{16}	P_{16}	PH_{16}

C   -   Conductometer

P   -   Potentiometer

pH   -   pH Meter

Col -   Colorimter

VIVA QUESTIONS

CONDUCTOMETRY

1. What is cell constant . what are its units ?

2. Mention the factors affecting conductivity of a solution ?

3. Name the type of cell and the electrodes used in conductivity cell ?

4. Why measured conductivity of Hcl is more than measured conductivity of CH_3COOH

5. Conductivity of Hcl Test Solution decreases when Naoh is added to it . Explain ?

6. Explain why conductivity increases after the end point in acid base conductometric titration ?

7. Represent the graph for (a) Strong Acid vs Strong Base   Conductometric Titration

   (b) weak Acid vs Strong Base

   (c) mixture of acid vs Strong Base

8. What is the change in conductivity when a solution is diluted ?

9. A water sample with high total dissolved solids (TDS) will have high or low conductivity ? give reason

COLORIMETRY

1. What is the range of visible radiation in (A $^\circ$ or nm)

2. Write the expression for Beer Lamberts Law ?

3. Write any three condition for beer – Lamberts law .

4. Why optical Density of Solution from Test tube no : 1 to Test tube no : 10 increases?

5. What is the role colored filters in colorimeter .

6. What is the relation between absorbance (A) and Transmittance (T) .

7. At the end of experiment , how can you say that Beer Lamberts Law is verified ?

8. Which of the following solutions can be estimated by colorimeter

   (a) Hcl   (b) $CuSo_4$ (c) NaoH   (d) $K_2Cr_2O_7$

9. A Solution shows > 0 % absorbance , transmittance = _____

10. Write the Statement of Beer Lamberts law .

POTENTIOMETRIC TITRATIONS

REDOX TITRATIONS

1. Name the electrodes in this experiment

2. Name the reference Electrode and write its electrode reaction

3. what is the role of salt bridge in the cell .

4. Represent the cell and indicate anode and cathode .

5. what is the use of plotting differential graph

6. calculate the emf of cell having .'

   $E^o{}_{SCE} = 0.242$ v   and $E^o{}_{Fe+2/Fe+3} = -0.44$ v

7. Write Nernst equation for potentiometric Redox Titration .

ACID – BASE TITRATIONS

1 . Represent the electrochemical cell for this titration .

2. Name the anode and cathode in this experiment .

3. Write the electrode reactions at anode and cathode

4. What is Quinhydrone electrode , what is its composition ?

5. What is E_{cell} in this titration ?

6. Represent and Explain the graph E_{cell} vs NaoH

7. What is the relation between E_{cell} and pH at neutralization point

8. Write any two advantages of Instrumental chemical analysis over volumetric analysis ?

pH – METRY

1. Represent the cell for pH metric acid base titration

2. What is pH , write the relation between P^H , write the Relation between P^H and $\{ H^+ \}$

3. What is pH Scale ? Explain .

4. What are the anode and cathode reactions in this experiment.

5. What is glass Electrode ?

6. Represent the graph pH vs V_{NaOH} and Explain

7. Why pH increases during titrations , and what is the change in E_{cell} . Explain ?

8. What is the appropriate value of pH and E_{cell} at neutralization point

9. What is a combined electrode

10. Write any two applications of pH measurements

11. What is the indicator electrode in this experiment . It is reversible to which ion ?