6. COURSE INFORMATION SHEET (CIS)

a) Course Description

COURSE TITLE	ENEERING CHEMISTRY LAB			
COURSE CODE	CH106BS/CH206ES			
REGULATION	R18-JNTUH			
COURSE STRUCTURE	LECTURES	TUTORIALS	PRACTICALS	CREDITS
	-	-	3	1.5
COURSE COORDINATOR	Dr. VENKANNA RAPOLU, ASSISTANT PROFESSOR			

b) Course Plan

Division of Experiments	List of experiments	Name of the Equipments	Course outcomes
Purity test of water	**Week-1:** Determination of total hardness of water by complexometric method using EDTA	--	CO1
	Week-2: Determination of chloride content of water by Argentometry	--	
Determination of acid content	**Week-3:** Estimation of an HCl by Conductometric titrations	Conductivity meter	CO2
	Week-4: Estimation of Acetic acid by Conductometric titrations r		
	Week-5 Estimation of HCl by Potentiometric titrations	Potentiometer	
Purity test of water	**Week-6:** Estimation of Fe^{2+} by Potentiometry using KMnO4		CO1

Kinetics of reaction	**Week-7** Determination of rate constant of acid catalysed hydrolysis of methyl acetate	--	CO3
Synthesis	**Week-8** Synthesis of Aspirin and Paracetamol	--	CO4
Separation of molecules	**Week-9** Thin layer chromatography calculation of Rf values. eg ortho and para nitro phenols	--	CO5
Determination of acid content	**Week-10** Determination of acid value of coconut oil	--	CO2
Physical adsorption	**Week-11** Verification of freundlich adsorption isotherm-adsorption of acetic acid on charcoal	--	CO6
Physical properties	**Week-12** Determination of viscosity of castor oil and ground nut oil by using Ostwald's viscometer.	Oswald's Viscometer	CO6
Separation of molecules	**Week-13** Determination of partition coefficient of acetic acid between n-butanol and water.	--	CO5
Physical properties	**Week-14** Determination of surface tension of a give liquid using stalagmometer.	Stalagmometer	CO6

c) Additional Experiments

Division of Experiments	List of experiments	Name of the Equipments	Course outcomes

d) Marks Distribution

Session wise marks	End semester exam	Internal marks
There shall be a continuous evaluation during the semester for 25 marks. Day-to-day work in the laboratory shall be evaluated for 15 marks and internal practical examination conducted by the concerned teacher shall be evaluated for 10 marks.	**75**	**25**

e) Evaluation Scheme

S .No.	Component	Duration	Marks
1	Day-to-day Evaluation	-	15
2	Internal Practical Examination	3hours	10
3	End Semester Examination	3 hours	75

f)**Text books & Reference books**

1. Senior practical physical chemistry, B.D. Khosla, A. Gulati and V. Garg (R. Chand & Co., Delhi)

2. An introduction to practical chemistry, K.K. Sharma and D. S. Sharma (Vikas publishing, N. Delhi)

3. Vogel's text book of practical organic chemistry 5th edition

4. Text book on Experiments and calculations in Engineering chemistry – S.S. Dara

7. MICRO LESSON PLAN

S.No.	Topic	Plan date	Actual date
1	Determination of total hardness of water by complexometric method using EDTA		
2	Determination of chloride content of water by Argentometry		
3	Estimation of an HCl by Conductometric titrations		
4	Estimation of Acetic acid by Conductometric titrations		
5	Estimation of HCl by Potentiometric titrations		
6	Estimation of Fe2+ by Potentiometry using KMnO4		
7	Determination of rate constant of acid catalysed hydrolysis of methyl acetate		
8	Synthesis of Aspirin and Paracetamol		
9	Thin layer chromatography calculation of Rf values. eg ortho and para nitro phenols		
10	Determination of acid value of coconut oil		
11	Verification of freundlich adsorption isotherm-adsorption of acetic acid on charcoal		
12	Determination of viscosity of castor oil and ground nut oil by using Ostwald's viscometer		
13	Determination of partition coefficient of acetic acid between n-butanol and water		
14	Determination of surface tension of a give liquid using stalagmometer		

8. LAB MANUAL

Experiment I

Determination of Total Hardness of Water by Complexometric Method Using EDTA

Aim: To estimate the total hardness of water by EDTA Method

Chemicals Required: Ammonia buffer solution, Eriochrome Black – T (EBT), Eriochrome Black – T (EBT), Magnesium chloride, EDTA Solution, Hard water

Apparatus: Conical Flask, Pipette, Burette, Beaker

Principle:

Ethylene diamine tetra acetic acid (EDTA) forms stable complexes with Ca^{+2} and Mg^{+2} ions present in water at pH 9-10. The sample of hard water must be treated with ammoncal buffer solution and EBT – indicator which forms unstable, wine red coloured complex with Ca^{+2} and Mg^{+2} present in water.

$$Ca^{+2} + EBT \rightarrow [Ca - EBT]$$

(Blue Colour) (Complex, wine red colour)

$$Mg^{+2} + EBT \rightarrow [Mg - EBT]$$

(Blue Colour) (Complex, wine red colour)

The stability of a metal indicator complex is less than that of metal EDTA complex. During the titration of the complex with EDTA, EDTA extracts the metal ions from the metal – ion – EBT indicator complex and forms stable, colorless complexes by releasing the free indicator. Where by the end point of the titration is the colour change from wine red to blue.

$$[(Ca^{+2} - EBT) + (Mg - EBT)] + EDTA \rightarrow (Ca - EDTA) + (Mg - EDTA)] + EBT$$

Wine red coloured complex Colorless & stable complex Blue in Colour

Procedure:

Preparation of standard hard water*:*

Dissolve one gram of pure, dry $CaCO_3$ in minimum quantity of diluted HCl and evaporate the solution to dryness on water bath. Dissolve the residue in small amount of water and transfer into a 100ml standard flask. Makeup the solution to the mark with distilled water and shake the flask well for uniform concentration.

Standardization of EDTA Solution:

Pipette out 10ml of standard hard water solution in to a conical flask, add 2 ml of buffer solution and 2-3 drops of EBT indicator and titrate the wine red coloured complex with EDTA solution taken in the burette, after rinsing the burette with EDTA solution, till the wine red colour of the solution changes to blue colour. Note the burette reading repeat the titration to get concurrent values.

Estimation of hardness of sample water:

Pipette out 20 ml of the water sample in to a 250ml conical flask, add 2ml of buffer solution and 3 drops of EBT – indicator. Titrate the wine red coloured solution with EDTA taken in the burette, till a clear blue coloured end point is obtained. Repeat the titration to get concurrent values.

Observation & Calculations

Standardization of EDTA:

S.No.	Volume of hard water	Burette reading		Volume of EDTA
		Initial	Final	
1				
2				
3				

Estimation of Hardness:

S.No.	Volume of Sample hard water	Burette reading		Volume of EDTA
		Initial	Final	
1				
2				
3				

Molority of standard Hard water $CaCO_3$

$$M_1 = \frac{Wt\ of\ CaCO_3}{M.wt\ of\ CaCO_3} \times \frac{1}{Volume\ of\ solution}$$

= _____ M

Molarity of EDTA Solution (M_2) =?

$$\frac{V1M1}{n1} = \frac{V2M2}{n2}$$

$n_1 = n_2 = 1$

$M_2 = \frac{V1M1}{V2}$ = _____ M

V_1 = Volume of standard hard water = _____ ml

V_2 = Volume of EDTA = _____ ml

M_1 = Molarity of standard hard water = _____ M

M_2 = Molarity of EDTA = _____ M

Molarity of the sample water = _____(M_3) or (M sample)

M_2V_2 = M_3V_3

M_3 = $\frac{M2V2}{V3}$ = _____M

V_2 = volume of EDTA

M_2 = Molarity of EDTA

V_3 = volume of hard water

Total hardness of water = M_3 x 100 x 1000

= _____ ppm.

Result:

The total hardness of sample of water = _____ ppm.

Experiment II

Determination of chloride content of water by Argentometry

Aim: Determine chloride ion concentration in a water sample by Argentometry (Mohrs method).

Chemicals required: Silver nitrate ($AgNO_3$), NaCl, Potassium chromate (K_2CrO_4) indicator.

Apparatus: Beaker, burette, pipette, conical flask, measuring jar, volumetric flask

Principle:

Chloride ion (Cl^-) is one of the major inorganic anions in water and wastewater. Along the sea coast chloride may be present in high concentration because of leakage of salt water into the sewage system. It also may be increased by industrial process. A high chloride contents may harm metallic pipes and structures as well as growing plants. The measured chloride ions can be used to know salinity of different water sources. For brackish water (or sea water or industrial brine solution), it is an important parameter and indicates the extent of desalting of apparatus required. Generally the Mohr's method is used to estimate the chloride present in water sample.

The Mohr method uses chromate ions as an indicator in the titration of chloride ions with a silver nitrate standard solution. After all the chloride has been precipitated as white silver chloride, the first excess of titrant results in the formation of a silver chromate precipitate, which signals the end point (1).

The reactions are:

$$Ag^+ + Cl^- \leftrightarrow AgCl(s)$$

$$2Ag^+ + CrO_4^{2-} \leftrightarrow Ag_2CrO_4(s)$$

Procedure:

Preparation of 5% K_2CrO_4 (indicator): Dissolve 1.0 g of K_2CrO_4 in 20 ml of distilled water.

Preparation of standard $AgNO_3$ solution: Transfer 9 g of $AgNO_3$ to a 500 ml volumetric flask and made up to volume with distilled water. The resulting solution was approximately 0.1 M.

Standardization of $AgNO_3$: Transfer 0.2500 g portions of NaCl into conical flask and dissolved in about 100 mL of distilled water. In order to adjust the pH of the solutions, add small quantities of $NaHCO_3$ until effervescence ceased. Add about 2 ml of K_2CrO_4 and titrate the solution to the first permanent appearance of red $Ag_2Cr_2O_4$.

Determination of Cl^- in solid sample: Take 20 ml of unknown water sample in to conical. Add small quantities of $NaHCO_3$ until effervescence ceased. Add about 2 ml of K_2CrO_4 and titrate the solution to the first permanent appearance of red Ag_2CrO_4.

Interpretation of data

Standardization of $AgNO_3$:

Replicate	Weight of NaCl (gr)	Volume of AgNO3 (ml)	Concentration of $AgNO_3$ (M)
1	0.25	V ml	M_1
2	0.25	V ml	M_2
3	0.25	V ml	M_3

Calculations for Replicate 1 of standardization:

Molecular mass of NaCl = 58.44 g/mole

M moles of $AgNO_3 = \frac{0.25\ gr\ NaCl}{58.44\ gr/mole} \times \frac{1000\ m\ moles\ NaCl}{1\ mole\ NaCl} = 4.278$ m moles

Molarity of $AgNO_3 = \frac{4.278}{V\ ml\ AgNO3} = M$

Molarity of $AgNO_3 = \frac{M1+M2+M3}{3} = M$

Determination of Chloride in Unknown:

Replicate	Volume of water sample	Volume of AgNO3 (ml)	Concentration of Cl^- solution
1	20 ml	V ml	
2	20 ml	V ml	
3	20 ml	V ml	

Atomic mass of Cl^- = 35.45 g/mole

M moles of Cl^- = $\mathbf{M}_{AgNO3}$ X $\mathbf{V}_{AgNO3}$ = M X V = x M moles

Mass of Cl^- = x M moles X 35.45 = y mg

Experiment III

Estimation of an HCl by Conductometric Titrations

Aim: To determine the strength of the strong acid by titration with strong base Conductometrically.

Chemicals Required: Sodium hydroxide, Hydrochloric acid

Apparatus: Conductivity Bridge, Conductivity cell, Burette, Beakers, Standard flask, pipette, Burette Stand.

Principle:

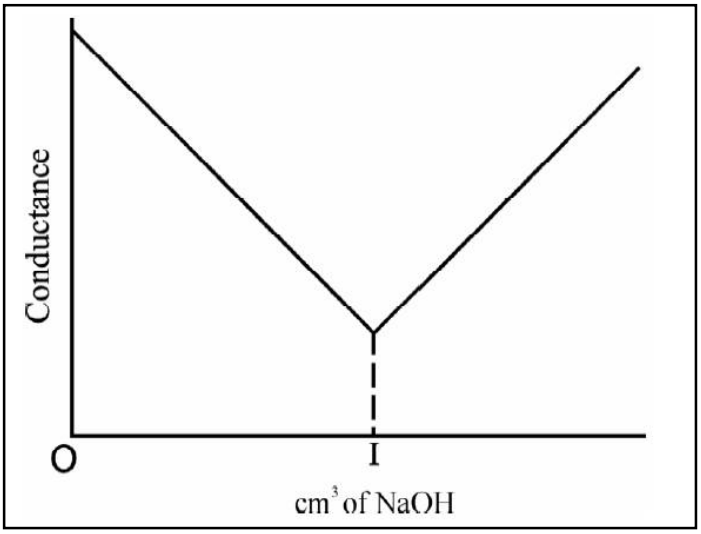
At first solution contain H+ and Cl^- ions. Since H^+ions posses greater mobility it follows that the conductivity is mainly due to H+ ions. The addition of NaOH is represented by the equation.

$H^+ + Cl^- + Na^+ + OH^- \longrightarrow Na^+ + Cl^- + H_2O$

As NaOH is added the H^+ ions are removed. The conductivity decreases as Na^+ ions do not process much mobility. As the neutralization point and solutions contains Na^+ions and Cl^- ions and will have minimum conductance value. If NaOH is further added this will add OH^- ions and so the conductivity increases.

Procedure:

Prepare a standard solution of 1M NaOH, and 0.1M HCl is prepared. 200 ml of HCl is taken in a 250 ml beaker. Further, the conductivity cell is washed with distilled water and rinsed with acid soln. The cell is kept in acid containing beaker and it is connected to the bridge. The conductivity of the solution is measured by adjusting the reading. NaOH solution is taken into burette and add 1 ml of solution to acid, stirred well and conductance is measured. Each time 1 ml of base is added to acid stirred well and the conductance is measured. For every instance equal numbers of values are taken on either side of the point of maximum. Repeat the procedure of addition of 1 ml NaOH and noting the conductivity of the resulting solution. Take 20-25 readings



Calculations:

Volume of unknown acid (V_1) =200 ml.

S. No	Volume of NaOH	Observed conductance
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		
16		
17		
18		
19		
20		

FORMULA:

$$N_1V_1\,(HCl) = N_2V_2\,(NaOH)$$

$$N_1 = \frac{1\ X\ V_2}{200}$$

Strength of solution = Eq. Weight X Normality

$$= 36.5\ X\ N_1$$

= ---------- g/lit

RESULT:

The normality of strong acid (HCl) determined by titrating against a strong base (NaOH) =_____N

Precautions:

1. The conductivity cell should be handled very carefully as it is very delicate.

2. Stirring should be done after each addition of the titrant.

Advantages or applications of conductance titration:

1. This method can be used to very dilute solutions.

2. Gives very accurate end points with an error of ± 0.5

3. These titrations are very useful in case of coloured solutions which cannot be titrated by ordinary volumetric method because colour change of indicator is not clear.

4. Useful of titrating weak acids against weak bases, which otherwise do not give sharp end points.

5. NO keen observation is necessary near the end point since it is detected graphically.

Experiment IV

Estimation of Acetic Acid by Conductometric Titrations

Aim: To determine the strength of the weak acid by titration with strong base Conductometrically.

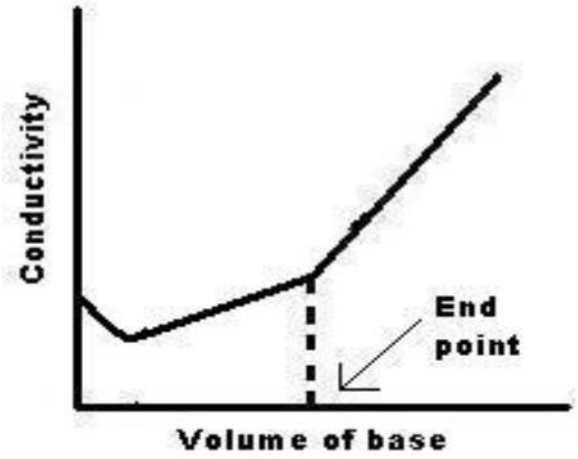
Chemicals Required: Sodium hydroxide, acetic acid

Apparatus: Conductivity Bridge, Conductivity cell, Burette, Beakers, Standard flask, pipette, Burette Stand.

Principle:

Before titration low initial conductance is observed due to low H^+ obtained during dissociation of weak CH_3COOH. During titration we can observe slight decrease of conductance due to consumption of H^+. During progress of titration we can observe slight increase in conductancedue to the presence of $CH3COO^-$ &Na^+ and nearly constant H^+ due to the buffer action of the produced CH_3COONa and the remaining CH_3COOH. After end point excess NaOH will lead to increase in conductance due to increasing of Na^+ and OH^-.

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



Procedure:

Prepare a standard solution of 1M NaOH. Similarly, prepare 0.1M acetic acid. 200 ml of acetic acid is taken in a 250 ml beaker. Further, the conductivity cell is washed with distilled water and rinsed with acid soln. The cell is kept in acid containing beaker and it is connected to the bridge. The conductivity of the solution is measured by adjusting the reading. NaOH solution is taken into burette and add 1 ml of solution to acid, stirred well and conductance is measured. Each time 1 ml of base is added to acid stirred well and the conductance is measured. For every instance equal numbers of values are taken on either side of the point of maximum. Repeat the procedure of addition of 1 ml NaOH and noting the conductivity of the resulting solution. Take 20-25 readings

CALCULATIONS:

Volume of unknown acid (V_1) =200 ml.

S. No	Volume of NaOH	Observed conductance
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		
16		
17		
18		
19		
20		

FORMULA:

$$N_1V_1\ (CH_3COOH) = N_2V_2\ (NaOH)$$

$$N_1 = \frac{1\ X\ V_2}{200}$$

Strength of solution = Eq. Weight X Normality

$$= 36.5\ X\ N_1$$

= ---------- g/lit

RESULT:

The normality of weak acid (acetic acid) determined by titrating against a strong base (NaOH) =_____N

Precautions:

1. The conductivity cell should be handled very carefully as it is very delicate.

2. Stirring should be done after each addition of the titrant.

Advantages or applications of conductance titration:

1. This method can be used to very dilute solutions.

2. Gives very accurate end points with an error of ± 0.5

3. These titrations are very useful in case of coloured solutions which cannot be titrated by ordinary volumetric method because colour change of indicator is not clear.

4. Useful of titrating weak acids against weak bases, which otherwise do not give sharp end points.

5. NO keen observation is necessary near the end point since it is detected graphically.

Experiment V

Estimation of HCl by Potentiometry

Aim: To determine the equivalence point between strong acid and strong base and to determine the normality of HCl by titrating with NaOH using potentiometer.

Chemicals required: 0.1N HCl, 1N NaOH, distilled water.

Apparatus: Potentiometer, standard cell, saturated calomel electrode, platinum electrode, beaker, burette, stirrer etc.

Principle

When a solution of strong acid (HCl) is titrated with the solution of a strong base (NaOH), the change in pH will be reflected in the change in EMF. When a small amount of alkali is added to the acid, a little change in the EMF is produced in the beginning. This change in electrode potential depends on the fraction of hydrogen ions removed. As an equivalence point is reached, the fraction of the hydrogen ions removed by constant volume of standard alkali increases rapidly, thereby causing a rapid change in the EMF. Thus if the EMF of the cell is plotted against the volume of the standard alkali added, a curve is obtained. As the change in EMF is much more rapid near the equivalent point, the exact equivalent point is obtained by differential method where, a graph of $\Delta E/\Delta V$ Vs. volume of alkali added, gives the maximum of the curve which corresponds to equivalence point of the titration.

$$HCl + NaOH \rightarrow NaCl + H_2O$$

The cell can be represented as

$$H_2(Pt) / acid\ solution / / KCl(aq) / calomel\ electrode$$

Procedure

Estimation of HCl:

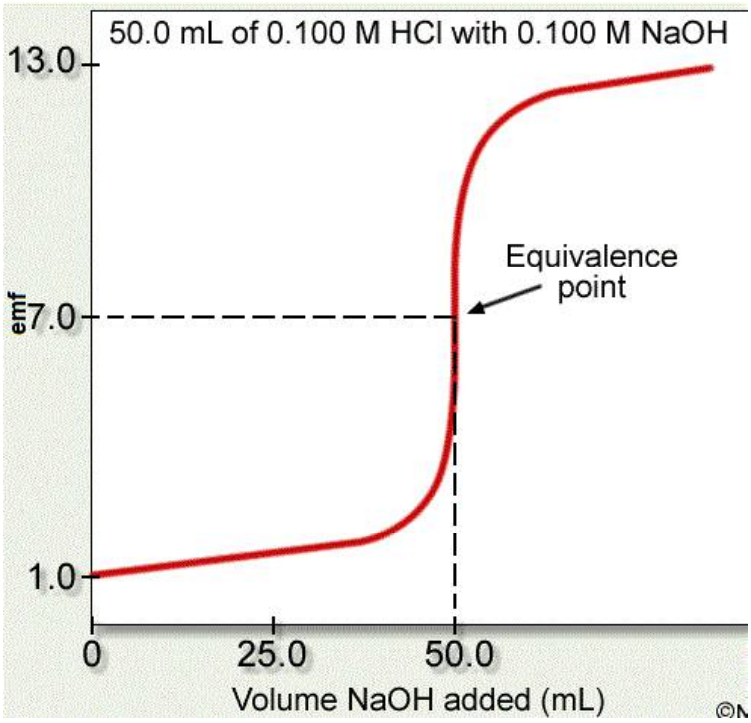
10 mL of 0.1N HCl is pipetted out into100 mL beaker and saturated it with Quinhydrone and the indicator electrode (platinum electrode) is dipped. The indicator electrode and saturated calomel electrode (reference electrode) are connected to the potentiometer. The two half cells are connected by means of a salt bridge. The potentiometer is standardized and used for measuring the emf directly. 1N NaOH is taken in the burette and is added to the HCl solution.

The emf of the acid, taken in the beaker, is measured initially. Rough titration is first carried out by adding 1 mL of NaOH and the emf for each addition is measured. The emf decreases gradually and then shows a sudden decrease in the emf. Enough readings are taken after the sudden decrease in emf. From these rough titrations, the range of end point is determined.

After finding the end point range, fair titrations are carried out by repeatedly adding 1 mL is added and emf is measured for each addition and the readings are tabulated.

Graph is plotted with volume of alkali (NaOH) along X-axis and measured emf along Y-axis. A sigmoid curve is obtained and the equivalence point is noted at the end point of intersection. To obtain a sharp end point, another graph of volume of alkali (NaOH) along X-axis and $\Delta E/\Delta V$ along Y-axis is plotted. The maxima obtained in the curve gives the accurate equivalence point.

Observations:



Pilot Titration:

Volume of alkali (NaOH) (mL)	EMF (mV)
0.0	
1.0	
2.0	
3.0	
4.0	
5.0	
6.0	
7.0	
8.0	
9.0	

Final Titration

Volume of alkali (mL)	EMF (mV)	$\Delta E/\Delta V$
0		
1.0		
2.0		
3.0		
4.0		
5.0		
6.0		
7.0		
8.0		
9.0		
10.0		
11.0		
12.0		
13.0		
14.0		
15.0		
16.0		

Calculations:

Strength of HCl

$Volume\ of\ acid\ taken\ in\ bea\,\text{ker}, V_1 = 20\,mL$

$Strength\ of\ acid, N_1 = ?$

$Volume\ of\ NaOH\ (\ from\ the\ graph), V_2 = ...mL$

$Strength\ of\ NaOH, N_2 = ...$

$According\ to\ Volumetric\ Law$

$V_1N_1 = V_2N_2$

$N_2 = \frac{V_1N_1}{V_2}N$

Amount of HCl

Amount of HCl present in the given solution=……(N_2)×36.45g

RESULT:

1. The equivalence point of potentiometric titration between strong acid Vs strong base is ... mL
2. The Normality of HCl by titrating with NaOH using potentiometer is……..N.
3. The amount of HCl present in the given solution =……g.

Experiment VI

Estimation of Fe^{2+} by Potentiometry using $KMnO_4$

Aim: To estimate the Fe^{2+} by Potentiometry using $KMnO_4$

Chemicals: $KMnO_4$, Distilled water, sulphuric acid

Apparatus: volumetric flask, pt electrode, saturated calomel electrode, potentiometer

Principle:

Potentiometric titration is the titration in which potentiometric measurements are carried out in order to fix the end point. In this method, the interest is with the change in electrode potential, rather than with an accurate value for the electrode potential in a given solution. In a potentiometric titration, the change in cell e.m.f. occurs most rapidly in the neighbourhood of the end point.

The Fe(II) –$KMnO_4$ redox system is represented as

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

The determining factor is the ratio of the concentrations of the oxidised and the reduced forms of the iron species.

For the reaction,

Oxidised form + ne- → Reduced form,

$$E = E^o + \frac{0.0591}{n} log \frac{[Ox]}{[Red]}$$

where E^0 is the standard Reduction Potential of the system. Thus the potential of the immersed electrode is controlled by the ration of these concentrations. During redox reactions, the potential changes more rapidly at the vicinity of the end point. The indicator electrode is usually a bright platinum wire or foil, the oxidising agent is taken in the burette. The cell can be represented as,

$$\text{Calomel} \parallel Fe^{3+}, Fe^{2+} \mid \text{Pt.}$$

Here Pt is the indicator electrode and calomel is the reference electrode.

Procedure:

Preparation of 0.1 N $KMnO_4$:

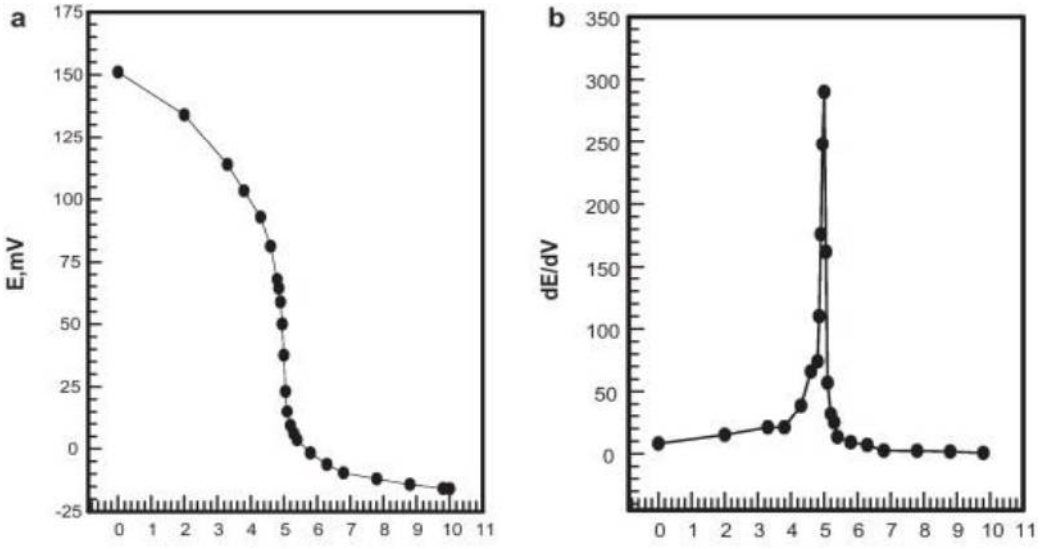
0.1 N $KMnO_4$ is prepared by dissolving 0.31 g of analar crystals in distilled water in a 100 ml volumetric flask. The solution is made up to the mark.

Calibration of the Potentiometer:

A standard cell of known emf is connected to the instrument and its emf is set in the voltage scale. The galvanometer key is pressed to complete the circuit and the deflection of the galvanometer needle is noted. If there is any deflection, the current passing through the rheostat is adjusted for null deflection. This procedure makes sure that the value of emf which is read on the scale is the true potential of the cell considered. The potentiometer is calibrated using the Weston standard cell of potential 1.018 V.

Estimation of Fe (Ii):

- The given Fe (II) solution is made upto 100 ml in volumetric flask.
- 20 ml of the solution is pipetted out into a clean beaker. To this, 25 ml of 2.5 M H_2SO_4 and 50 ml of distilled water are added.
- A platinum electrode is dipped into this solution, and it is coupled with a calomel electrode through a salt bridge. The resulting cell is connected to the potentiometer.
- Standard $KMnO_4$ solution is added from the burette, to this solution, insteps of 1 ml and the emf is recorded after each addition.
- At the end point, there is a jump in emf due to the absence of Fe^{2+}. The approximate range of the end point is determined.
- The experiment is repeated by adding the titrant in steps of 0.1 ml near the end point. A graph is plotted between emf, E and the volume of dichromate added. The inflexion point gives the volume of titrant at the end point.
- The first derivative ($\Delta E/\Delta V$ vs. Volume of titrant) curves give the exact volume of dichromate required for the reaction. From the plot of E vs. Volume of titrant, potential at the equivalent point is obtained.
- Atomic weight of Fe is 55.85



Volume of $KMnO_4$

Table:

S.No	Volume of $KMnO_4$	EMF	ΔE	$\Delta E/\Delta V$	V_{mean}
1					
2					
3					
-					
-					

Volume of $KMnO_4 = V_1$

Concentration of $KMnO_4 = N_1 = 0.1$ N

Volume of Fe^{2+} solution = $V_2 = 20$ ml

Concentration of Fe^{2+} solution = $N_2 = ?$

According volumetric law $N_1V_1 = N_2V_2$

$N_2 = N_1V_1/V_2$

Amount of Fe^{2+} ion present in the given solution= (N_2 x Equivalent weight of Fe^{2+}) /10

= (N_2 x 55.85/10) gm

Result: The amount of Fe^{2+} ion present in the given solution=…….gm

Experiment VII

Determination of Rate Constant of Acid Catalyzed Hydrolysis of Methyl Acetate

Aim: To determine the rate constant of the hydrolysis of Methyl acetate using an acid as a catalyst.

Chemicals: Methyl acetate, distilled water, HCl, NaOH, Phenolphthalein indicator, 0.1 N oxalic acid

Apparatus: Conical flask, volumetric flask, burette, pipette, reagent bottles.

Principle:

Methyl acetate undergoes hydrolysis, in the presence of an acid (HCl, for example), to give acetic acid and methyl alcohol.

$$CH_3COOCH_3 + H_2O \rightarrow CH_3COOH + CH_3OH$$

In the presence of an acid, this reaction should be of second order, since two molecules are reacting. But, it is found to be first order. This may be explained in the following way : The rate of the reaction is given by

$$dx / dt = k^1 [CH3COOCH_3] [H_2O]$$

where k' is the rate constant (or specific rate constant). Since water is present in large excess, its active mass (molar concentration) virtually remains constant during the course of the reaction. Therefore, its active mass gets included in the constant, and the above equation reduces to:

$$dx / dt = k^1 [CH_3COOCH_3]$$

Thus, the rate of the reaction is determined by one concentration term only (that is, by a single power of the concentration term only). Hence, the reaction is first order. Such reactions are also referred to as pseudo first order reactions. The progress of the reaction (hydrolysis of ester) is followed by removing a definite volume of the reaction mixture, at definite intervals of time, cooling it in ice, and titrating the acetic acid formed against alkali, which has already been standardized. The amount of alkali used is equivalent to the total amount of hydrochloric acid present originally and the amount of acetic acid formed in the reaction.

The amount of acetic acid formed (x), at definite intervals of time (t), can be obtained. The amount of acetic acid formed, at the end of the reaction, is equivalent to the initial concentration (a) of the ester. Suppose the volumes of the sodium hydroxide solution (titre value) required for neutralization of 5 ml of the reaction mixture are :

(i) at the commencement of the reaction is Vo

(ii) after time (t) is Vt

(iii) at the end of the reaction is V_∞

Then:

x (amount of acetic acid formed after time) is proportional to (Vt -Vo).

a (initial concentration of ester) is proportional to (V∞ -Vo).

[a – x] (concentration of ester present after time t) is proportional to

(V∞ -Vo) - (Vt -Vo) = (V∞ -Vt)

The first order rate expression given by :

$$k_1 = \frac{2.303}{t} \log \frac{a}{[a-x]}$$ would correspond to :

$$k_1 = \frac{2.303}{t} \log \frac{(V_\infty - V_o)}{(V_\infty - V_t)}$$

Hence, the rate constant (k_1) could be calculated.

Procedure

Step I:

Standardization of NaOH using standard Oxalic acid (0.1N)

1. Pipette out 10ml of given 0.1N standard Oxalic acid into a 100ml conical flask.

2. Titrate this solution against the given unknown concentration of NaOH using phenolphthalein indicator until the end point is colorless to pale pink.

3. Tabulate the values and repeat the titration for concurrent readings and determine the unknown concentration of supplied NaOH solution.

Table 1

S.No	Volume of Oxalic Acid taken (ml)	Burette Readings (ml) Initial	Final	Volume of NaOH consumed (ml)
1				
2				
3				

$$N_1V_1 = N_2V_2$$

Here, N1 = concentration of oxalic acid, V_1= Volume of oxalic acid

N_2 = concentration of NaOH, V_2= Volume of NaOH

Step II:

Standardization of HCl using NaOH solution

1. Pipette out 2 ml of given HCl into a 100ml conical flask.

2. Titrate this solution against the NaOH using phenolphthalein indicator until the end point is colorless to pale pink.

3. Tabulate the values and repeat the titration for concurrent readings and determine the unknown concentration of supplied HCl solution.

Table 2

S.No	Volume of HCl taken (ml)	Burette Readings (ml) Initial	Final	Volume of NaOH consumed (ml)
1				
2				
3				

(NaOH) N_2 V_2 = N_3 V_3 (HCl)

Concentration of HCl, N_3 = _____

Step III:

Determination of rate constant (k1) for the acid-catalyzed hydrolysis of methyl acetate

1. Take 100 ml of given HCl (whose strength is determined in step II) solution in a stoppered reagent bottle.

2. Add 5 ml of methyl acetate solution to the HCl solution. Note the time when half of the methyl acetate solution is added. The mixture is shaken well.

3. Pipette out 5 ml of the reaction mixture and discharge it into 50 ml of ice cold water kept in a conical flask.

4. Titrate the reaction mixture against NaOH solution using phenolphthalein as indicator. This titre value corresponds to Vo.

5. Repeat steps 3 and 4 at intervals of 5, 10, 15, 20, 30, 45, 60 minutes. Each titre value corresponds to Vt.

6. Take the remaining solution in a stoppered conical flask and heated to 60^{o}C, and keep at this temperature for 5 minutes.

7. The solution is allowed to cool to room temperature.

Table 3

S.No	Time (min)	Volume of solution taken (ml)	Burette Readings (ml) Initial	Final	Volume of NaOH consumed (ml)	$(V_\infty - V_t)$ (ml)	$\log(V_\infty - V_t)$	$k_1 = \frac{2.303}{t} \log \frac{(V_\infty - V_o)}{(V_\infty - V_t)}$ (min^{-1})
1	0 (V_0)							
2	5 (V_{t1})							
3	10 (V_{t2})							
4	15 (V_{t3})							
5	20 (V_{t4})							
6	30 (V_{t5})							
7	45 (V_{t6})							
8	60 (V_{t7})							
9	V_∞							

8. Repeat Steps 3 and 4. This titre value corresponds to V_∞ till concurrent values are obtained.

9. Plot a graph of log (V_∞ -Vt) versus time (t) and determine the slope.

10. Report the theoretical and graphical value of rate constant (k_1).

Observations and Calculations:

Room Temperature = ……..... ^oC ; V_∞ = …….. ml ; V_o = …….. ml ; (V_∞-Vo) = …….. ml ;

log(V_∞-Vo) = ...

Mean Value of Rate Constant (k1) = _____

Results:

1. Strength of NaOH Solution = _____

2. Strength of HCl Solution = _____

3. Rate Constant (k_1) for the acid-catalyzed hydrolysis of methyl acetate at …^oC = =_____ (theoretical).

= _____ (graphical).

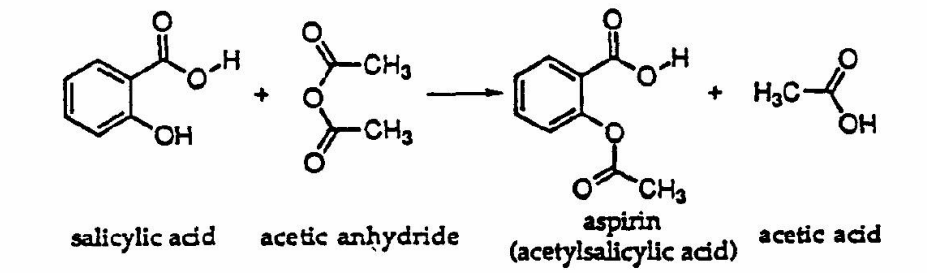
Experiment VIII

Synthesis of Aspirin and Paracetamol

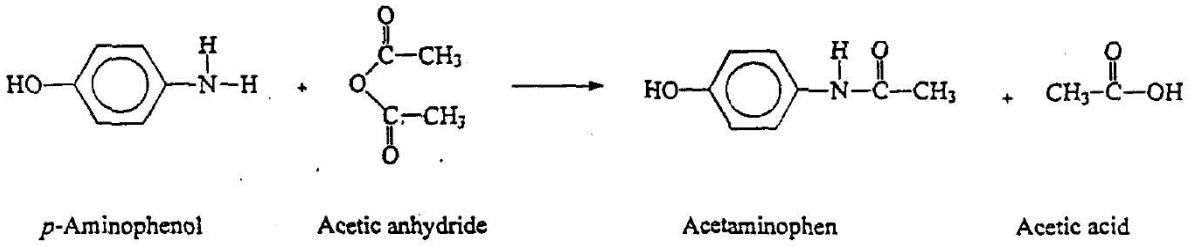
Aim: To synthesize some common pain relievers: aspirin and paracetamol

Principle: Aspirin (acetylsalicylic acid) is both an organic ester and an organic acid. It is used extensively in medicine as a pain killer (analgesic) and as a fever-reducing drug (antipyretic). When ingested, acetylsalicylic acid remains intact in the acidic stomach, but in the basic medium of the upper intestinal tract, it hydrolyzes forming the salicylate and acetate ions.

Aspirin (molar mass of 180.2 g/mol) is prepared by reacting salicylic acid (138.1 g/mol) with acetic anhydride (molar mass of 102.1 g/mol). Aspirin is a weak monoprotic acid.



Paracetamol (acetaminophen) acts as a fever reducer and pain reliever. Acetaminophen is an amide, a compound that is a derivative of ammonia that has been reacted with an acidic substance, in this case, acetic acid. It can be found in several analgesic preparations, such as Tylenol, some of which may contain other ingredients such as caffeine and buffers.



PROCEDURE

a) Preparation of Aspirin

Chemicals: Salicylic acid, acetic anhydride, sulfuric acid, ethanol, ice.

Apparatus: Dropper, Erlenmeyer flask 125 mL, Beakers, Graduated cylinders, Watch glass, Stirring rod, Ring stand, Buchner funnel, filter paper to fit Buchner funnel, vacuum filtration flask, Rubber tubing for vacuum flask, thermometer, dropper.

Procedure

- Weigh out 2.0 g of salicylic acid. Place it in a 125 mL conical flask. Add 5 mL of acetic anhydride.

- Swirl the flask to wet the salicylic acid crystals. Add 5 drops of concentrated sulfuric acid, to the mixture.
- Gently heat the flask in a boiling water bath for about 10 minutes.
- Remove the flask from the hot water bath and add 10 ml of deionized ice water to decompose any excess acetic anhydride.
- Chill the solution in an ice bath until crystals of aspirin no longer form, stirring occasionally to decompose residual acetic anhydride.
- If oil appears instead of a solid, reheat the flask in the hot water bath until the oil disappears and again cool.
- Set up a vacuum filtration apparatus. Wet the filter paper in the Buchner funnel with 1-2 mL of distilled water.
- Turn on the water aspirator. Decant the liquid onto the filter paper, minimizing any transfer of the solid aspirin.
- Add 15 mL of cold water to the flask, swirl, and chill again. Pour the liquid and the crystals of aspirin onto the filter paper. Repeat until the transfer of the crystals to the vacuum filter is complete.
- Determine the mass of the crude aspirin crystals.

b) Preparation of Paracetamol (Acetaminophene)

Chemicals: Acetic anhydride, Phosphoric acid, Ethanol

Apparatus: Dropper Erlenmeyer flask, 125 mL Beakers, Graduated cylinders, Watch glass Stirring rod,, Vial to hold aspirin sample, Ring stand Clamp (to hold 125-mL conical flask) Buchner funnel, Filter paper to fit Buchner funnel, Vacuum filtration flask, Rubber tubing, Ice, Dropper.

Procedure:

- Fill a 400 mL beaker about half full with water. Place the beaker and water on a hot plate and bring to a boil.
- Weigh out 1.5 g of p-aminophenol and transfer it into a 125 mL conical flask. (Avoid contact with skin. You may wish to wear gloves.)
- Add 25 mL of water. Add 20 drops of concentrated phosphoric acid (H_3PO_4), and swirl the flask until all of the amine dissolves. If not, add a few more drops of phosphoric acid.
- Turn off the hot plate. Place the flask in the hot water. Carefully add 2 mL of acetic anhydride to the flask. Leave the flask in the warm water for 10 minutes.
- Remove the flask and place it an ice-water bath. Stir the mixture to crystallize the acetaminophen. You may need to scratch the walls of the flask to start the crystallization. If no crystals appear, add a small seed of acetaminophen to start the crystal formation. Allow the flask to stay in the ice-water bath for 30 minutes.
- Collect the crystals in a Buchner funnel using vacuum filtration. Wash the crystals with 10 mL of cold water. Allow the crystals to dry.

- Determine the mass of the crude acetaminophen.

Results:

Mass of salicylic acid = …… gms

Mass of crude aspirin = …… gms

Yield of aspirin = ……. %

Mass of P-amino phenol = …… gms

Mass of paracetamol = …… gms

Yield of paracetamol = …….. %

Experiment IX

Thin Layer Chromatography Calculation of Rf Values. Eg Ortho and Para Nitro Phenols

Aim: To calculation the R_f Values of ortho and para nitro phenols by using thin layer chromatography.

Chemicals: O-nitro phenol, P-nitro phenol, ethyl acetate, di-chloromethane

Apparatus: TLC plate, capillary tubes,

Principle:

Most reactions produce more than one product. Naturally occurring materials are only rarely 100% pure. It is therefore desirable to have a simple, fast and efficient way to determine the purity of Organic mixtures. The separation of a mixture by passing it, in solution, over an adsorbent (such as Alumina or Silica Gel) is the basic idea of Chromatography. It involves the passage of a mobile phase across a stationary phase in a column. Usually a mixture of compounds is present in the mobile phase. As soon as the mixture comes in contact with the stationary phase, some or all of the components of the mixture are adsorbed on it. As additional mobile phase comes along, some or all of the mixture will dissolve and continue moving. This adsorption/solution process continues along the length of the column. If a proper choice of mobile phase, stationary phase, solvent and other operating parameters was made, the mixture will be separated in the column and its various components will emerge at different times.

In Thin Layer Chromatography, a liquid solution is directly applied to a solid adsorbent. Capillary action draws a developing solvent up the TLC plate. As this solvent passes through the spot, the mixture will be dissolved and will begin to move with the solvent front. However, the adsorbent will also reabsorb part or all of the mixture. As more solvent comes by, the mixture will again go into solution, move further and be reabsorbed. Since different materials will be dissolved and reabsorbed at different rates, separation will take place. This passage of the solvent front through the adsorbent is known as **developing** the plate. The extent of separation, measured by retention factor ("R_f ") value differences, will depend on the relative solubilities and relative strengths of adsorption of the components of the mixture. Organic compounds interact with absorbents by a variety of interactions. If the compound is non-polar, it can only have weak 'Van der Waals' attractions for the absorbent. However, more polar molecules may interact more strongly by a variety of mechanisms including dipole-dipole interactions, coordination, and hydrogen bonding. The most important rule of chromatography is that *the more polar compounds will be absorbed most strongly on absorbents (stationary phases), while non-polar compounds will be only very weakly absorbed.* In a typical chromatography experiment, the non-polar compounds, since they are poorly absorbed, will be held least strongly and will move quickly through the plate. Polar compounds, on the other hand, will be slowed on their process

through the plate by their strong interactions with the solid phase. This separation based on polarity will explain most of the chromatography encountered in this course.

Types of Adsorbents used in Chromatography

Listed in decreasing power of adsorption:

Alumina > Activated Charcoal > Magnesium Silicate > Silica > Starch

Solvents Commonly Used in Chromatography

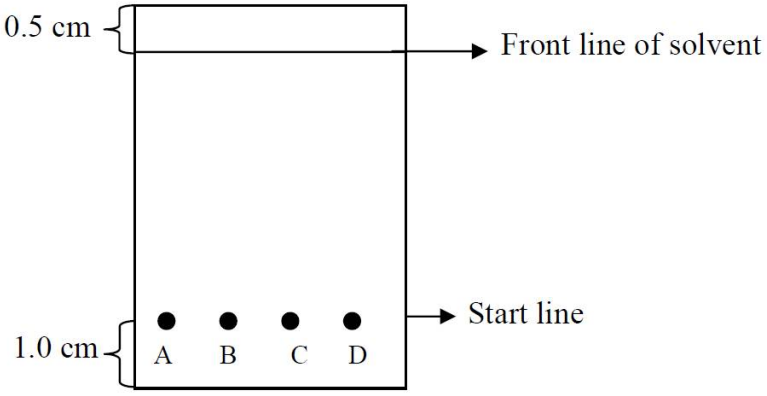
Listed in decreasing polarity:

Acetic Acid >Water > Methanol > Acetone > Ehtyl acetate > Diethyl ether > Chloroform > Methylene chloride > Toluene > Cyclohexane > Petroleum ether

For a typical separation, a variety of different combinations of solvent and adsorbent may be effective. Once you have developed your plate, it must be **visualized**. This visualization may be accomplished by reacting the developed plate with a chemical reagent. Iodine (I_2) is one of the easiest to use of the several common chemical visualizing agents. The developed slide is simply exposed to I_2 vapors in a chamber similar to the developing chamber for a few minutes. Almost all compounds will form a weak colored complex with the I_2. This complex will appear as a darker area on the slide. The 'spots' are characterized by their R_f value, a measure of how far the spot traveled with that combination of adsorbent and solvent.

Procedure:

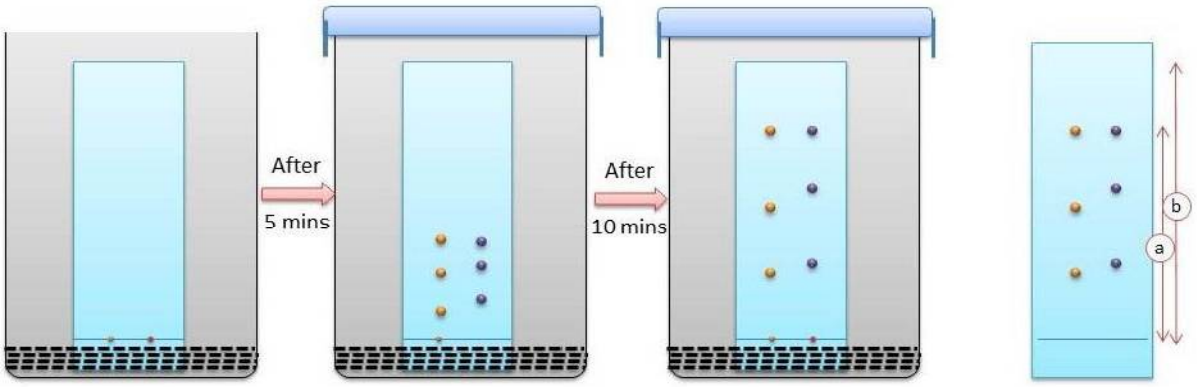
- Take 1 TLC plate handle it only on the edges, as fingerprints contain UV-active materials.
- Using a pencil draw a very light line across the sheet (short dimension) about 1 cm from one end.
- Then make 4 small light marks at even intervals along the line for spotting the samples. Draw another light line about 1 cm from another end of the plate for the solvent front.
- Obtain a TLC chamber and place solvent, a 5% ethyl acetate in dichloromethane to 0.5 cm height.
- Place a piece of filter paper around the inside surface of the container and extend into the solvent.
- A glass jar with a lid or a beaker with a watch glass or a cover of a Petri dish can be used as a TLC chamber:
- Using clean capillary tubes carefully spot four samples at two pencil marks as shown below.
- The spots should be as small as possible in order to minimize tailing and overlapping when the TLC plate is developed. If a more intense spot is desired, let the spot dry and re-spot in the same location.
- When the spots are dry, place the TLC plate in the developing chamber. Then gently close the chamber.



A: o-Nitrophenol B: p-Nitrophenol

- Be sure that the bottom edge of the TLC plate is in the solvent but the spots are above the solvent, and the filter paper does not touch the chromatographic sheet.
- When the solvent has moved to the front line, remove the plate. Lay it on a clean surface in well ventilated area and allow the solvent to evaporate until the plate appears dry.
- Visualize the plate under iodine chamber Measure all the distances traveled by the compounds and solvent.
- Calculate the retention factor (R*f*) for each compound. and immediately draw a light pencil line around each spot.

Calculation of retention factor:



$$Rf = \frac{\text{distance travelled by the component}}{\text{distance travelled by the solvent}} = \frac{a}{b}$$

Retention factor $Rf = \dfrac{\text{Distance travelled by the component}}{\text{Distance travelled by the solvent}}$

Result:

Retention factor of o-nitro phenol = ……

Retention factor of p-nitro phenol = ……

Experiment X

Determination of Acid Value of Coconut Oil

Aim: To Determine the Acid Value of Coconut Oil

Chemicals: NaOH, distilled water, Phenolphthalein indicator, potassium hydrogen phthalate, coconut oil, ethanol, diethyl ether

Apparatus: Volumetric flask, conical flask pipette**,** burette, weighing balance, beaker

Principle:

Acidity may occur in coconut oil upon storage due to the decomposition of the oil to free fatty acids, peroxides and low molecular weight aldehydes and ketones, which produce distinctive smell and affect the quality of oil. Acid value may be defined as the number of milligrams of KOH required to neutralise the free acid in 1gm of fatty oil.

Procedure:

Preparation of Solutions:

1) **0.1 N Sodium hydroxide:** Weigh accurately 0.4gm of sodium hydroxide into 100mL standard flask, dissolve in little distilled water and make up the solution to the mark with distilled water and shake the flask well for uniform concentration.
2) **Ethanol-ether solution:** Prepare a mixture of ethanol and diethyl ether (1:1 v/v); add 1mL of phenolphthalein indicator and titrate with NaOH till pale pink colour is observed. This is the neutralisation of ethanol-ether solution.
3) **Standard solution of potassium hydrogen phthalate:** Accurately weigh about 1.02gm of potassium hydrogen phthalate into 100mL flask dissolve in little water make up the solution to the mark with distilled water.

Determination of acid value:

1) **Standardisation of NaOH solution:** Pipette out 20mL of potassium hydrogen phthalate solution into a 250mL conical flask and add 2 drops of phenolphthalein indicator and titrate the solution with NaOH taken in burette. Pale pink colour is the end point of the titration. Note the burette reading and let the value be x mL
2) **Standardisation of coconut oil:** Accurately weigh 1gm of coconut oil into a 250mL conical flask; add 50mL of ethanol-ether solution. Shake the flask well for uniform dissolution of the sample. Add 1 drop of phenolphthalein indicator and titrate the solution with NaOH taken in the burette till pale pink colour is observed. Note the burette reading, let the value be y mL

Calculations:

(M_1) Molarity of potassium hydrogen phthalate = $\frac{1.2 \text{ X } 1000}{204.22 \text{ X } 100} = 0.058M$

(M_2) Molarity of NaOH: M_1V_1/M_2V_2

$M_2 = M_1V_1/x$

Acid value of coconut oil = $\frac{M_{NaOH}}{W}$ (W = wight of coconut oil = 1 gm)

Result: The acid value of given oil is ……..

Experiment XI

Verification of Freundlich Adsorption Isotherm-Adsorption of Acetic Acid on Charcoal

Aim: To study the adsorption of acetic acid from aqueous solution by activated charcoal & examine the validity of Freundlich & Langmuir's isotherm.

Chemicals: 0.1N acetic acid solution, 0.1N sodium hydroxide solution, phenolphthalein indicator, powdered activated charcoal etc.

Apparatus: Five reagent bottles with stoppers, burette, pipette, conical flasks

Principle: Adsorption is accumulation of a substance at an interface. The adsorption of a solute from a solution, generally follows the Freundlich empirical adsorption isotherm given by

$$\frac{x}{m} = KC^{\frac{1}{n}}$$

Where

x = weight of adsorbent, m = mass of adsorbent, K = constant representing the capacity of the adsorbent, and C = equilibrium concentration of the solution.

From the concentration (C), the volume of each original solution and concentration of each solution after equilibrium (Ce), the weight of oxalic acid per gram of the adsorbent (x/m) is calculated as follows.

$$\frac{x}{m} = \frac{(C - C_e)\ X\ 60}{10}$$

Draw a graph of log (x/m) vs. log Ce and calculate the constant n and K using the equation.

$$log\frac{x}{m} = log\ K + \frac{1}{n} log C_e$$

Procedure:

1. Take five clean, dry stopper reagent bottles & label them from 1 to 5.
2. Add by means of a burette 50, 40,30, 20 &10 ml of 0.1N acetic acid solution & 0, 10, 20, 30 & 40 ml distilled water in bottle nos. 1,2,3,4 & 5 resp.
3. Weigh accurately 1 gm of activated charcoal & add to bottle no.1. Similarly, add 1 gm activated charcoal in each of the remaining bottles. Stopper the bottles & shake them well.
4. Meanwhile determine the initial concentration of acid by titrating against std. NaoH solution using phenolphthalein indicator.

5. Using dry filter papers of dual size filter the solution of each bottle in separate dry flasks. Titrate 10 ml of filtrate from each bottle with 0.1Nn NaOH using phenolphthalein indicator. Stake three readings for each bottle & take the mean. These readings gives the equilibrium concentrations of the solution i.e. concentration of acid after adsorption. Tabulate the result.

Observation table:

Bottle No.	Initial conc. of acid C_0 m eq./lit	Equilibrium Conc. of acid , Ce gm eq./lit	Amount of acid adsorbed (x)	x/m	log x/m	log Ce
1						
2						
3						
4						
5						

Here $x = \frac{(C0 - Ce) \times V \times \text{Eq.wt.of the acid}}{1000}$

Where, V = total volume (50ml) of the solution in each bottle.

Calculation:

Calculate initial concentration (C_0) of acetic acid in gm equivalent per lit for each sample. Also calculate the equilibrium concentration (Ce) of the acid solution in each of the bottles in gm equivalent per lit. Then calculate the amount of the acid adsorbed in each bottle as follows:

Graph:

1. Plot the graph of log(x/m) (Y-axis) against log Ce (X-axis). A straight line will be obtained

This is in agreement with Freundlich equation. Find slope & Y-intercept values.

2) Plot Ce /(x/m) (Y-axis) against Ce (X-axis). A straight line obtained shows the agreement with Langmuir adsorption isotherm. Calculate slope & Y-intercept.

Result:

Thus Freundlich & Langmuir adsorption isotherm are studied by the above Experiment.

Experiment XII

Determination of Viscosity of Castor Oil and Ground Nut Oil by Using Ostwald's Viscometer

Aim: To determine the viscosity of the given castor oil and ground nut oil

Chemicals: astor oil and groundnut oil, distilled water.

Apparatus: Specific gravity bottle, viscometer, rubber tube with screw pinch cock, stand, beaker

Theory: The force of friction which one part of the liquid offers to another part of the liquid is called viscosity. For measuring the viscosity coefficient, Ostwald viscometer method is used which is based on Poiseuille's law. According to this law, the rate of flow of liquid through a capillary tube having viscosity coefficient 'η' can be expressed as

$$\eta = \frac{\pi \cdot r^4 tP}{8vl}$$

where, v= vol. of liquid (in ml)

t= flow time (in sec.) through capillary

r= radius of the capillary (in cm)

l= length of the capillary (in cm)

P= hydrostatic pressure (in dyne/sq.cm)

η= viscosity coefficient (in poise).

Since, the hydrostatic pressure (the driving force) of the liquid is given by P = dg h (where h is the height of the column and d is the density of the liquid);

$$\eta \alpha P t \text{ ; or, } \eta \alpha dght$$

If, η_1 and η_2 are the viscosity coefficients of the liquids under study, d_1, d_2 are their densities and t_1 and t_2 are their times of flow of *equal volume* of liquids through the same capillary respectively, then

$$\eta_1 \alpha d_1 g h t_1$$

$$\eta_2 \alpha d_2 g h t_2$$

Hence

$$\frac{\square_1}{\square_2} = \frac{\square_1 \, \square_1}{\square_2 \, \square_2}$$

Here, usually the viscosity of given liquid is measured with respect to water whose viscosity is known very accurately at different temperatures. The SI physical unit of viscosity is the pascal-second (**Pa·s**), (i.e., kg·m−1·s−1). This means: if a fluid with a viscosity of one **Pa·s** is placed

between two plates, and one plate is pushed sideways with a shear stress of one pascal, it moves a distance equal to the thickness of the layer between the plates in one second. The cgs unit for the same is the **poise** (P), (named after J. L. Marie Poiseuille). It is more commonly expressed, as **centipoise** (**cP**). [1 cP = 0.001 Pa·s]. Water at 20 °C has a viscosity of 1.0020 cP.

Procedure:

1. Note the laboratory temperature.

2. Wash the density bottle with distilled water and dry.

3. Take the weight of the empty & filled (with distilled water) specific gravity bottle (with stopper). Then, weigh the filled with specific gravity bottle unknown given liquids individually. Use the data for measuring the densities.

4. Clean and rinse the viscometer properly with distilled water. Fix the viscometer vertically on the stand and filled with specific amount (say 20ml) of mixture (every time take the same volume).

5. Time of flows was recorded for each solution (water and the given liquids).

6. Take 3 to 4 readings.

Observations:

1. Laboratory temperature=…. ^{0}C

2. Density measurement:

Weight of empty density bottle (w1) =……..g.

Weight of density bottle with water (w2) =……..g.

Weight of density bottle with castor oil (w3) =…….g.

Weight of density bottle with castor oil (w4) =…….g.

So,

Weight of water = (w2-w1) =…g.

Weight of castor oil = (w3-w1) =…g.

Weight of ground nut oil = (w4-w1) =…g

Sample	**Flow times**			
	t_1	**t_2**	**t_3**	**Average**
Water				
Castor oil				
Ground ut oil				

Calculations:

Determination of the viscosity of the liquid (η)

As discussed warlier

$$\frac{\square_{\square}}{\square_{\square}} = \frac{\square_{\square}\ \square_{\square}}{\square_{\square}\ \square_{\square}}$$

$$\eta_c = \frac{\square_{\square}\ \square_{\square}}{\square_{\square}\ \square_{\square}} \text{ x } \square_{\square}$$

here $\eta_c =$ viscosity of castor oil; $\eta_w =$ viscosity of water (1.0020 cP at 20^0C)

d_c = density of castor oil; dw = density of water

t_c = flow time for castor oil; t_w = flow time for water

$$\frac{\square_{\square}}{\square_{\square}} = \frac{\square_{\square}\ \square_{\square}}{\square_{\square}\ \square_{\square}}$$

$$\eta_g = \frac{\square_{\square}\ \square_{\square}}{\square_{\square}\ \square_{\square}} \text{ x } \square_{\square}$$

here $\eta_c =$ viscosity of ground nut oil; $\eta_w =$ viscosity of water

d_c = density of ground nut oil; dw = density of water

t_c = flow time for ground nut; t_w = flow time for water

Result:

The viscosity of the given castor oil with respect to water at laboratory temperature was found to be ……cP.

The viscosity of the given groundnut oil with respect to water at laboratory temperature was found to be ……..cP.

Experiment XIII

Determination of Partition Coefficient of Acetic Acid between N-Butanol and Water

Aim: To determine the partition coefficient of acetic acid between n-butanol and water

Chemicals: Acetic acid, n-butanol, distilled water

Apparatus: Stoppard bottle, beaker, pipette, conical flask

Principle:

In dilute solutions at constant temperature a solute which exists in the same molecular species in two non-miscible solvents, will distribute itself between these two solvents at constant temperature according to the partition law, the partition coefficient:

$k = C_1/C_2$

where

C_1 = concentration of acetic acid in water, and

C_2 = concentration of acetic acid in buty1 alcohol

The concentrations C_1 and C_2 must be expressed in the same units either as grams, gram molecules, or gram equivalents per litre.

Procedure:

- Boil about 200 mL of distilled water in a beaker for 10 minutes. Pour into a flask and stopper lightly and cool. This is CO_2 free water for later use.
- In a 200 mL glass stoppered bottle place about 70 mL of approximately 2M acetic acid and 50 mL of n-butyl alcohol.
- Stopper the bottle and shake well for at least 1 minute, and then allow the liquid layers to separate. Note and record the temperature of the mixture.
- Insert a 25 mL pipette and carefully withdraw a 25 mL aliquot of the upper alcohol layer. The pipette should first be rinsed by sucking up a little of the solution and discarding this.
- Pipette the solution into a second glass stoppered bottle and add an approximately equal volume of boiled distilled water to this second bottle.
- Shake-well to transfer the acid to the water layer, add 3 drops of phenolphthalein and titrate with the approximately 1.0 M sodium hydroxide. The bottle should be stoppered from time to time and vigorously shaken and titration continued until a faint permanent pink colour remains.
- Pipette, also a 25 mL aliquot from the lower aqueous layer of the first bottle as follows. Close the pipette with the finger and place it carefully in the lower layer.
- Suck up and blow out gently a small quantity of liquid to wash out any small quantity of the upper layer that has got into the pipette. Rinse the pipette with a little of solution.

- Allow the solutions to settle and withdraw 25 mL of the lower solution. Place this solution in a flask, add three drops of phenolphthalein and titrate with the approximately 1.0 M sodium hydroxide.
- Add about 25 mL each of fresh butanol and boil distilled water (but no further acid) to the original mixture remaining in the first bottle. Repeat the procedure above and sample and titrate with the new concentrations of acid.

The following table summarises the above procedure:

Initial mixture: 50 mL n-butyl alcohol, 70 mL 2M acid:

Summary of procedure

Alcohol Layer		Water Layer		Alkali for Titration mL		K = C1/ C2
Sample Removed mL	Fresh Alcohol Added	Sample Removed mL	Water Added mL	Water Alcohol C1	C2	
25	--	25	--			
--	25	--	25	--	--	
25	--	25	--			

Result: The partition coefficient of acetic acid between n-butanol and water is……

Experiment XIV

Determination of Surface Tension of a Given Liquid Using Stalagmometer

Aim: Determine the surface tension of a given liquid at room temp using stalagmometer by drop number method.

Apparatus: Stalagmometer, specific gravity bottle, a small rubber tube, screw pinch cork

Chemicals: Distilled water, experimental liqui

Principle: In the drop number method, the number of drops formed by equal volumes of two liquid is counted. If m_1 and m_2 is the mass of one drop of each of the liquid having densities d_1 and d_2 respectively. If n_1 and n_2 is the number of drops formed by volume v of the two liquids, then their surface tensions are related as

$$¥_1/¥_2 = (d_1/d_2)*(n_2/n_1)$$

One of the liquid is water its surface tension and density are known. Then the surface tension of the given liquid can be calculated.

Procedure:

1. Clean the stalagmometer with chromic acid mix, wash with water and dry it

2. Attach a small piece of rubber tube having a screw pinch cock at the upper end of the stalgmometer.

3. Immerse the lower end of the stalagmometer in distilled water and suck the water 1-2cm above mark A. adjust the pinch cork so that 10-15 drops fall per minute .

4. Clamp the stalgmometer allow the water drops to fall and start counting the number of drops when the meniscus crosses the upper mark A and stop counting when the meniscus passes mark B

5. Repeat the exercise to take three to four readings

6. Rinse the stalgmometer with alcohol and dry it

7. Suck the given liquid in the stalgmometer and count the drops as in case of water

8. Take a clean dry weighing bottle weighs it with water as well as with liquid.

9. Note the temp of water taken in a beaker.

Observations:

Room temp = t^0C

Density of water = d_w

Surface tension of water = ¥ dynes/cm

No of drops From a Fixed Volume				Mean
Liquid	1….	2……	3…..	n_l=
Water	1….	2…….	3……	n_w=

Weight of empty specific gravity bottle = $w_1$gram

Weight of specific gravity bottle + water = w_2 gram

Weight of empty sp.gravity bottle + liquid = $w_3$gram

Weight of water= (w_2-w_1) gram

Weight of liquid= (w_3-w_1) gram

Calculations:

Density of the liquid

D_l= (w_3-w_1)/(w_2 –w_1) *d_w

Surface tension of liquid =

$¥_l$ = (d_l/d_w)*(n_w/n_l)* $¥_w$

Result

The surface tension of liquid is ……dynes/cm.

9. VIVA QUESTION AND ANSWERS

1. Why should weights not be lifted with hand ?

Ans. This causes error because some matter may be transferred from the hand to the weight.

2. What is end point ?

Ans. The stage during titration at which the reaction is just complete is known as the end point of titration.

3. Why a titration flask should not be rinsed ?

Ans. This is because during rinsing some liquid will remain sticking to the titration flask therefore the pipetted volume taken in the titration flask will increase.

4. What are primary and secondary standard substances ?

Ans. A substance is known as primary standard if it is- available in high degree of purity, if it is stable and unaffected by air, if it does not gain or lose moisture in air, if it is readily soluble and its solution in water remains as such for long time.On the other hand, a substance which does not possess the above characteristics is called a second-ary standard substance. Primary standards are crystalline oxalic acid, anhydrous Na CO , Mohr's salt, etc.

5. Burette and pipette must be rinsed with the solution with which they are filled, why ?

Ans. The burette and pipette are rinsed with the solution with which they are filled in order to remove any substance sticking to their sides, which otherwise would decrease the volume of the liquids to be taken in them.

6. It is customary to read lower meniscus in case of colourless and transparent solutions and upper meniscus in case of highly coloured solutions, why?

Ans. Because it is easy to read the lower meniscus in case of colourless solutions, while the upper meniscus in case of coloured solutions.

7. What is a normal solution ?

Ans. A normal solution is a solution, a litre of which contains one gm-equivalent of the solute. This is symbolised as 1 N.

8. Why the last drop of solution must not be blown out of a pipette ?

Ans. Since the drops left in the jet end is extra of the volume measured by the pipette.

9. Pipette should never be held from its bulb, why ?

Ans. The heat of our body may expand the glass bulb and introduce an error in the measurement of the volume.

10. What is acidimetry and alkalimetry ?

Ans. It is the volumetric analysis involving chemical reaction between an acid and a base.

11. What do you mean by 1.0 M solution ?

Ans. A solution containing 1 mole of solute per litre of solution is 1.0 M solution.

12. Which indicator is used in the titration of sodium carbonate against hydrochloric acid and what is the colour change at the end point ?

Ans. Methyl orange. The colour change is yellow to pinkish red.

13. What is the difference between an end point and an equivalence point ?

Ans. End point is the point at which the indicator shows a visible change indicating that the reaction has completed. Equivalence point is the point at which stoichiometric amounts of the two reactants have been added. Visible end point may or may not exactly coincide with equivalence point**.**

14. What indicator is used in the titration of oxalic acid with sodium hydroxide ? Which solution is taken in the burette and what is the end point ?

Ans. Phenolphthalein. Sodium hydroxide solution is taken in the burette. Appearance of pink colour is the end point.

15. What is the indicator used in the titration of sodium carbonate against hydrochloric acid ? Which solution is taken in the burette and what is the end point ?

Ans. Methyl orange. Acid solution is taken in the burette, change of colour from yellow to pink is the end point.

16. What is basicity of an acid ?

Ans. It is the number of replaceable hydrogen atoms in a molecule of the acid.

17. What is the relation between equivalent mass of acid and its molecular mass ?

Ans:

$$Equivalent \quad mass \quad of a \quad acid = \frac{Molecular mass}{Basicity}$$

18. What is acidity of a base ?

Ans. It is the number of OH ions furnished by a molecule of the base.

19. What is the relation between equivalent mass of a base and its molecular mass ?

Ans.

$$Equivalent \quad mass \quad of a \quad base = \frac{Molecular mass}{Acidity}$$

20. Why hot liquids should not be taken in the Burette?

A. Hot liquids should not be placed in the burette because the instrument is calibrated at a much lower temperature (15 to 20^{o} C).

21. Which meniscus is read in case of colored solution taken in a burette?

Ans. In case of colored solution the top of the meniscus read on the burette scale as lower part is not visible.

22. What are the requirements of a standard substance?

Ans. A standard substance (primary standard) is required to fulfill the following conditions:

i. It must be in a highly purified state.

ii. It must be stable in air

iii. It should be readily soluble in water.

23. What is redox titration?

Ans. The reactions which involve simultaneous oxidation and reduction are called redox titrations and the titrations involving redox reactions are called redox titrations.

24. What is Permanganometry?

Ans. Redox titrations involving $KMnO_4$ as the oxidizing agent are called Permanganometry.

25. Why is dilute H_2SO_4 must suitable as compared to HCl and HNO_3 in Potassium permanganate titrations?

Ans. HCl reacts with $KMnO_4$ to liberate Cl_2 gas and consumes some KMnO4, higher results are obtained. HNO_3 is a stronger oxidizing agent than KMnO4, so it will oxidize Fe^{+2} to Fe^{+3} so lower results of the titration will be obtained.

26. Why does $KMnO_4$ act as self indicator?

Ans. $KMnO_4$ solution is purple in color due to presence of MnO4- ions. In presence of dil. H_2SO_4 it reacts with reducing agents (Fe^{+2} or Oxalic acid) and gets reduces to Mn^{+2} ions. So the color disappears. At the end point, when all the reducing agent has been oxidized, the excess drop of $KMnO_4$ added is not reduced and pink color is observed in the solution. The color is light pink since solution is very dilute.

27. In $KMnO_4$ titration $KMnO_4$ should be added in small lots, why?

Ans. If KMnO4 is added in larger amount, a brownish precipitate of hydrated MnO2 formed which interferes in getting the correct end point. The same appears if dil. H2SO4 is not added before the titration.

28. Why only a very small excess of $SnCl_2$ is added during the reduction of Fe^{+3} ions?

Ans. If large excess of $SnCl_2$ is added during reduction, a much larger amount of $HgCl_2$ will have to be added to destroy excess of $SnCl_2$. Further a very thick ppt of Hg_2Cl_2 will be formed, which reacts very slowly with $K_2Cr_2O_7$ solution.

29. What is oxidation state of Cr in $K_2Cr_2O_7$?

A. +6

30. Is KMnO4 a primary standard? Give reason for your answer.

Ans. KMnO4 is not a primary standard because it cannot be obtained in a high state of purity. It contains small amount of MnO2 as impurity.

31. What are Iodine titrations?

Ans. The redox titrations using iodine directly or indirectly as an oxidizing agent are called iodine titration.

32. What do you understand by Iodometric titration?

Ans. Iodine titration in which some oxidizing agents liberate iodine from KI and then liberated iodine is titrated against standard solution of reducing agent added from a burette.

33. Name some oxidizing agents which can be titrated by iodometry?

Ans. Acidified CuSO4, acidified K2Cr2O7, KMnO4, Fe+3 ion, Cl2,Br2 etc.

34. Why hypo is commonly used as a reducing agent in iodine titrations?

Ans. Hypo is preferred to other reducing agents in iodometry because it is a primary standard.

35. Which indicator is used in iodine titrations and what is color change at the end point

Ans. Freshly prepared starch solution is used as an indicator. Starch gives blue color with iodine. Just disappearance of blue color is the end point of these titrations.

36. What are complexometric titrations?

Ans. Titrations depending upon the combination of ions, other than H+ or OH-, to from a soluble slightly dissociated complex ion or compound are called complexometric titrations.

37. Name the most important complexing agent employed in complexometry?

Ans . Ethylene diamine tetraacetic acid (EDTA)

38. What form of EDTA is used in titrametric analysis?

Ans. Used as the disodium salt of EDTA

39. Give some examples of titrations involving EDTA as a complexing agents?

Ans. Estimation of temporary and permanent hardness of water and estimation of metal ions such as Cu+2, Ni+2, Zn+2 and Mg+2 etc.

40. Which type of ligand is EDTA?

Ans. It serves as a hexadentate ligand and acts as a chelating agent.

41. Name the most widely used indicator in EDTA titrations. How does it act?

Ans. In EDTA titrations the commonly used indicator is Erichrome Black T called Ero=T. It shows a color change from red to blue in the pH range 7-11.

42. At what pH the hardness of water is estimated by EDTA method? How this pH is maintained?

Ans. The pH value is adjusted to about 10 by using a buffer solution of NH4Cl and NH4OH. At higher pH values CaCO3 or Mg(OH)2 may get precipitated and the indicator may change its color. At lower pH values, Mg- indicator complex becomes unstable and a sharp end point cannot be obtained.

43. What is a buffer solution?

Ans. A buffer solution is defined as a solution which resists any change in its pH value even when small amounts of the acid or the base are added to it. A buffer solution gives acidic as well as basic ions in solution which destroy the excess of any acid or base added keeping the pH constant.

44. What is an acidic buffer? Give an example.

Ans. It is solution of a mixture of weak acid and salt of weak acid with a strong base.(Ex. CH3COOH+CH3COONa)

45. What is a basic buffer? Give an example.

Ans. It is the solution of a mixture of a weak base and a salt of this weak base with a strong acid (Ex. NH4OH + NH4Cl).

46. What is conductance and what are its units?

Ans. The reciprocal of resistance is called conductance. Its units are ohm^{-1} or mho or Siemens (S)

47. What is specific conductance and what are its units?

Ans. Specific conductance is defined as the conductance of one centimeter cube (1c.c.)of the solution. It is denoted by K (kappa)

Specific conductance = Cell constant/ observed resistance.

Its units are $Ohm^{-1}cm^{-1}$ or mho cm^{-1} or S cm^{-1}

48. What is a conductivity cell?

Ans. The vessel in which the measurement of conductivity of the solution is to be made is known as conductivity cell. They are of various shapes and sizes depending upon the nature of the solution taken.

49. Why ordinary water is unsuitable for conductivity measurements? What is conductivity water?

Ans. Ordinary water is unsuitable for conductivity measurements because it posseses large conductance due to the material dissolved from the container and due to CO_2 and NH_3 dissolved from air. So, water is specially purified by distilling it a number of times after addition of a little

$KMnO_4$. Such water is known as conductivity water and should have a conductivity not more than 2-3 $X10^{-8}$ ohm $^{-1}$.

50. What are the advantages of conduct metric titrations?

Ans. Conduct metric titrations have a number of advantages over volumetric titrations involving the use of indicators:

a. These titrations can be used for colored solutions where ordinary indicators fail to give the end point.
b. These can be used for the titration of even very dilution solutions of the order of 10-4 M.
c. No extra care is needed near the end point as it is simply the intersection of two lines.
d. These can be used for the titration of mixtures of weak and strong acids.

51. Name three electrodes, which are usually employed to measure pH of a solution. Which one is most suitable?

Ans. Hydrogen electrode, Quinhydrone electrode and glass electrode. Glass electrode is the most suitable for this purpose.

52. What is a combined electrode? What is the mechanism of its working?

Ans. Electrode combining reference electrode and a glass electrode is termed as combined electrode. These are convenient to use and are common these days. The schematic of the cells formed may be represented as:

$^{+}$Ag, AgCl, |experimental solution| KCl (Salt solution) Hg2Cl2(s) Hg Pt $^{-}$

The glass electrode acts as –ve electrode and the calomel electrode acts as +ve electrode.

53. Why is hydrogen electode not generally used in pH measurements ?

Ans. i. it is difficult to set up.

ii. It cannot be used in redox system.

iii. It cannot be used in the presence of Hg, As, S, Fe+3, MnO4 etc.

54. Why are solutions of acids and bases in water acts as Type equation here. electrolytes?

Ans. 5hey consist of hydrated ions and therefore conduct electricity.

55. Define a reference electrode?

Ans. It is a Standard hydrogen electrode. It is obtained by dipping a platinum foil in 1M HCl solution through which hydrogen gas is passed at 25^{o}C at 1atm pressure. Its electrode potential is taken as 0.

56. Why glass electrode is preferred to quinhydrone electrode in measuring pH of solution.

Ans. Glass electrode is simple, not easily oxidized and attains equilibrium rapidly. It can be used safely up to pH 10. Wheras quinhydrone electrode can be used upto pH 8, it cannot be used in redox solution.

57. What is an electrochemical cell?

Ans. It is a device to convert the chemical energy of a redox reaction into electrical energy by bringing about the redox reaction indirectly in two separate halves.

58. What is electrode potential?

Ans. The tendency of an electrode to lose or gain electrons, when it is in contact with its own ions.

59. Define reduction potential?

Ans. The tendency of electrode to lose electrons, when it is in contact with solution of its own ions.

60. Define oxidation potential?

Ans. The tendency of electrode to gain electrons, when it is in contact with solution of its own ions.

61. Define a reference electrode?

A. An electrode whose electrode potential is accurately knows or whose electrode potential has been arbitrarily fixed.

62. What is a fuel cell?

Ans. A device for converting the energy of fuel directly into electrical energy.

63. Why do electrochemical cells stop working after sometime?

Ans. An electrochemical cell produces electrical energy at the cost of redox reaction. When the redox reaction is completed, the cell stops working.

64. What is function of salt bridge in a cell?

Ans. It completes the circuit. Moreover it maintains neutrality of the solutions in the two half cells.

65. What is cell constant? give its units.

Ans. It is the ratio between the distance of two parallel plates of the cell and the area of the electrode. Its unit is cm-1.

66. State Beer=Lambert's Law.

Ans. The absorbance (A) is directly proportional to the molar concentration (C) as well as path length

(l) i.e. $A \alpha Cl$ or $A = \epsilon Cl$, where ϵ is molar absorptivity coefficient. Mathematically, the law can also be stated as:

$$I_t = I_o 10^{-\epsilon Cl}$$

67. What is colorimetry?

Ans. The method of analysis which involves the measurement of absorption of light radiations in the visible region of the spectrum is called colorimetry.

68. What is the basis of colorimetry?

Ans. The variation of the color of the solution with change in concentration of the ions forms the basis of colorimetry.

69. What is visible spectrum? What happens when visible spectrum is made to fall upon a colored solution?

Ans. The visible region of spectrum is considered to extend from 3800-7800Å. When a beam visible light passes through a colored solution contained in a tube, absorption of radiation energy take place and there is loss in energy of the radiation. The emerging radiation is always less energetic than the entered one.

70. What is a calibration curve? How is it drawn?

Ans. Calibration curve is a graph between optical densities (absorbance) against concentration. It will be straight line for those solution which obey Beer's law .using standard solution of different concentration, a calibration's curve for a suitable solute in solution is drawn.

71. How can we select proper wavelength for given solution?

Ans. The absorbance of the given solution at different wavelength is measured. The plot of absorbance verses its wavelength gives λ max, which is the suitable wavelength for the colorimetric determination of the given solution.

72. Name the types of instruments employed in absorption measurements?

Ans. There are three types:

i. Colorimeter.

ii. Absorption meter.

iii. Spectrophotometer.

73. What is the difference between a colorimeter and spectrophotometer?

Ans. Colorimeter determines the concentration of a substance by measurement of relative absorption of light. In such instrument the absorption in the visible region is generally employed.