North Maharashtra University, Jalgaon.

M.Sc.
Physical Chemistry Practical

Part-I

Practical Procedures

From June 2015

Dear friends,

It gives immense pleasure for putting forth that experimental procedure in Physical Chemistry of M. Sc. (I) which thoroughly discussed and finalized in the Work Shop at S. P. D. M. College Shirpur on 10/07/2015. It will be definitely useful for teachers and students.

I extend my words of thanks to Dr. C.P.Sawant (Chairman, BOS in Chemistry) who participated in the workshop and puts valuable words towards the efforts taken. Also thankful to Prof. B.V.Pawar (Dean Science faculty) and authorities of North Maharashtra University for permission of workshop.

I would like to thanks to the teachers who attended the workshop and shared their views for preparing these procedures. Dr. V. T. Patil (Z. B. Patil College, Dhule) V. S. Pawar (Z. B. Patil College , Dhule), Dr. V. S. Zope (M. J. College, Jalgaon), R. S. Saralkar (Pratap college, Amalner), Jyoti Mahashabde(R. C. Patel College, Shirpur) P. B. Wagh (Jijamata College, Nandurbar), Dr. A. M. Nemade (G. D. Bendale Mahila College, Jalgaon), S. P. Patil (G. T. P. College, Nandurbar), Dr. U. M. Jadhav (P. S. G. V. P. College, Shahada), N. I. Shaikh (P. G. C. S. T. R. , Jalgaon), R. D. Talele (D. N. College, Faizpur), Mrs. J. U. Shinde, (M. J. College Jalgaon), Devendra Dhami (R. C. Patel, College, Shirpur), M. C. Chaudhari (R. C. Patel, College, Shirpur)

I would like to thanks to my colleagues of Chemistry Dept S.P.D.M . College, Shirpur for preparing the booklet of my procedure. My special thanks to Mrs. Sarojbala Mahire ,P.G.Patil, V.B. Mahale, L.P.Chaudhari, N.B.Jadhav, Dr. K.R. Desale , M.A. Patil, Y.V.Satkar ,and J.A. Patil.

I would like to accept every responsibility for any error which may be skipped off due to little span of time.

Dr. S.N.Patel
Co-ordinator of Workshop.
Member of BOS

SEM -I

INSTRUMENTAL:-

Conductometry:-

1) Determination of degree of hydrolysis and hydrolysis constant of sodium acetate conductometrically.

2) Determination of the concentration of sulphuric acid, acetic acid and copper sulphate by conductometric titration with sodium hydroxide.

Potentiometry:-

1) To determine the stability constant of a complex ion $[Ag\ (S_2O_3)]^{-3}$ potentiometrically.

2) To determine standard free energy change ΔG^0 and equilibrium constant for the reaction $Cu + 2Ag^+ = Cu^{+2} + 2Ag$ potentiometrically.

pH -metry:-

1) Determination of Hammett constant of a given substituted benzoic acid by pH measurements.

2) To determine the amount of aspirin in the given tablet.

Colorimetry/ Spectrophotometry:-

1) To determine pKa and Ka of given indicator by colorimetry/ spectrophotometry.

2) To determine the empirical formula of Ferric salicylate complex by Job's method and verify by slope ratio method.

Surface Chemistry

1) To determine CMC of the given surfactant (Sodium Lauryl Sulphate) by surface tension method.

2) To determine the critical micelle concentration of soap by surface tension method.

NON INSTRUMENTAL:-

Chemical kinetics:-

1) To determine the rate constant for depolymerization of diacetone alcohol catalyzed by sodium hydroxide using dilatometer.

2) Kinetics of reaction between potassium persulphate and KI. Determination of rate constant; study of influence of ionic strength.

3) To determine the order of the reaction between potassium persulphate and potassium iodide by fractional change method.

Non instrumental:-

1) Determine the transport number of H^+ and Cl^- ions by moving boundary method.

Conductometry

<u>**Aim:-**</u>

Determine the degree of hydrolysis and hydrolysis constant of sodium acetate conductometrically.

<u>**Chemicals:-**</u>

0.1 N sodium acetate, 0.1 N acetic acid, 0.02 N KCl

<u>**Procedure:-**</u>

1. Note down the conductivity value of 0.02 N KCl Solution.

2. Prepare 0.1N, 0.05 N and 0.025 N solution of Sodium acetate in water and note down their conductivities.

3. Prepare 0.1N, 0.05 N and 0.025 N solution of Sodium acetate in 0.1N Acetic acid and note down their conductivities.

4. Calculate the cell constant using formula.

$$\text{Cell constant } (K_{cell.}) = \frac{\text{Specific conductance of 0.02 N KCl solution } (L_s)}{\text{Observed Conductance of the same KCl solution } (L)}$$

5. Calculate specific conductance (K_c) at different concentrations of Sodium acetate in water and Sodium acetate in 0.1N Acetic acid.

6. Calculate equivalent conductance (λ_c) at different concentrations of

   Sodium Acetate in water (λ_c) and Sodium acetate in 0.1N Acetic

   acid ($\lambda_c^{'}$).

$$\lambda_{eq.} = \frac{1000 \times K_c}{C}$$

7. Calculate degree of hydrolysis of Sodium acetate using formula.

$$h = \frac{(\lambda_c - \lambda_c')}{(\lambda_c'' - \lambda_c')}$$

8. Calculate hydrolysis constant (K_h) using formula.

$$K_h = \frac{h^2 C}{(1-h)}$$

Where C = Concentration of Sodium acetate.

Observation and calculation :-

1. Conductance of 0.02 N KCl solution = _____ mhos.
2. Equivalent Conductance of NaOH at infinite dilution (λ") = 238

Concentration of Sodium acetate (M)	Solvent	Specific Conductance (K_c)	Equivalent conductance (λ_c)	Degree of hydrolysis (h)	Hydrolysis constant (K_h)
0.1 0.05 0.025	Water				
0.1 0.05 0.025	Acetic acid				

Result:- 1) Degree of hydrolysis of sodium acetate=_____

2) Hydrolysis constant of sodium acetate=_____

<u>Aim:-</u>

To determine the concentrations of H_2SO_4, CH_3COOH and $CuSO_45H_2O$ in a mixture by conductometric.titration with sodium hydroxide

<u>Chemicals :-</u>

0.1 N H_2SO_4 solution, 0.1 N CH_3COOH solution ,

0.1 N $CuSO_45H_2O$ solution and 0.25 N NaOH solution

<u>Procedure:-</u>

1) Pipette out 5 ml of H_2SO_4, 5 ml of CH_3COOH and 5 ml of $CuSO_45H_2O$ solution in a 100 ml beaker.
2) Add 35 ml of distilled water in it.
3) Dip conductivity cell in it and connect it to Conductivity Bridge.
4) Measure the resistance/conductance of the solution.
5) Fill the microburette with standard solution of NaOH
6) Add 0.2 ml of 0.25 M NaOH solution from the microburette into the mixture, stir and measure the resistance/conductance of the solution.
7) Plot conductance Vs ml of NaOH added.
8) Present your observations as follows :

No.	ml of NaOH added	R (ohm)	Conductance ohm^{-1}
1	0.0		
2	0.2		
3	0.4		
4	0.6		
5	0.8		
.	.		
.	.		

Calculation:-

There are three break observe in the graph. Draw the perpendicular from these three intersection points and note down the volume of NaOH require to neutralize the H_2SO_4, CH_3COOH and $CuSO_45H_2O$ solution.
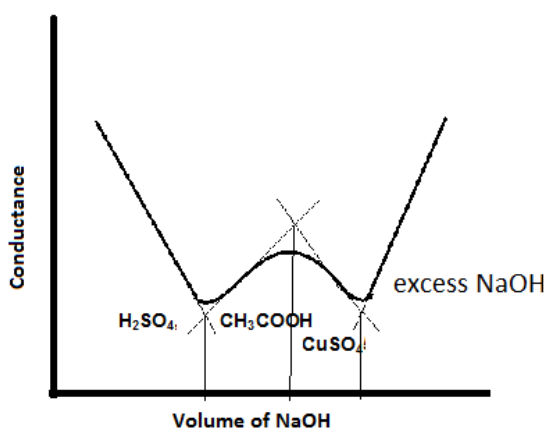
Let V_1, V_2, V_3 are the values of alkali corresponding to first , second and third break respectively.

V_1 ml corresponds to H_2SO_4.

$(V_2- V_1)$ ml corresponds to CH_3COOH.

(V_3-V_2) ml corresponds to $CuSO_45H_2O$.

1. Calculate normality and hence strength of H_2SO_4.
2. Calculate normality and hence strength of CH_3COOH.
3. Calculate normality and hence strength of $CuSO_45H_2O$.

Result:-

1. Normality of H_2SO_4.=---------------------------
2. Strength of H_2SO_4. = -------------------------------
3. Normality of CH_3COOH.=----------------------------
4. Strength of CH_3COOH. = -------------------------------
5. Normality of $CuSO_45H_2O$.=----------------------------
6. Strength of $CuSO_45H_2O$. = -------------------------------

Potentiometry

Aim:-

Determine the stability constant of the complex ion Ag $(S_2O_3)_2^{3-}$ potentiometrically.

Apparatus:-

Potentiometer Assembly, two silver electrode, KNO_3 salt bridge, Volumetric Flask numbered 1to5 , burette, pipette 50ml beaker

Chemicals :-

0.01M AgNO3 solution, 0.1M $Na_2S_2O_3$ solution,

Procedure:-

1) Prepare five different concentrations of the complex as shown in table.

Flask No.	1	2	3	4	5
AgNO$_3$ solution (ml)	5	5	5	5	5
Na$_2$S$_2$O$_3$ solution (ml)	10	15	20	25	30

Make up the volume of each flask to 50 ml with distilled water.

2) Standardize the potentiometer.
3) Pipette out 10 ml of the 0.01M $AgNO_3$ in a 50 ml beaker and dip in it a clean polished silver electrode
4) Pipette out 25 ml of the complex ion solution from the flask no. 1 in another 50 ml beaker. Dip in it a clean polished silver electrode
5) Connect the two half cell by salt bridge to form the concentration cell as $^{(-}$ $^)$Ag$_)$ / Ag$^+$ ion in complex // 0.01 M AgNO$_3$ (0.01M) / Ag$^{(+)}$
6) Connect the cell to the potentiometer
7) Measure the EMF of the concentration cell
8) Repeat the step 4to7 for all remaining complex solution

Flask No.	Emf of cell(E)	Initial conc.		Equilibrium conc.			K$_s$
		AgNO$_3$ (C1)	Na$_2$S$_2$O$_3$	Ag$^+$ (C$_2$)	(S$_2$O$_3$)$^{2-}$	Complex ion	
1		0.001 M	0.02 M		0.018 M	0.001 M	
2		0.001 M	0.03 M		0.028 M	0.001 M	

3		0.001 M	0.04 M		0.038 M	0.001 M	
4		0.001 M	0.05 M		0.048 M	0.001 M	
5		0.001 M	0.06 M		0.058 M	0.001 M	

Calculations :-

i)   Calculate the equilibrium concentrations of Ag^+ ions (C_2) from the emf of the cell as $E = 0.0592 \log_{10}. C_1/C_2$ where $C_1 = 0.01M$

ii)   Calculate the concentration of complex ion:

     [complex ion]= Initial concentrations of $AgNO_3$, [complex ion]= 0.001 M

iii)   Calculate the concentration of free $(S_2O_3)^{2-}$ ion:

     $[(S_2O_3)^{2-}]$ (free)= $[(S_2O_3)^{2-}]$ (initial) = $2[Ag(S_2O_3^-)^2]$

     Since for one mole of the complex two moles of $(S_2O_3)^{2-}$ ions are consumed

     $[(S_2O_3)^{2-}] = [(S_2O_3)^{2-}]$ (initial)_0.002

iv)   The formation of complex is represented as

     $Ag^+ + 2[(S_2O_3)^{2-} \rightarrow Ag(S_2O_3^-)_2$

     The stability constant of the complex ion is given by

$$K_s = \frac{[complex\ ion]}{[Ag^+]\ [(S_2O_3)^{2-}]}$$

$$K_s = \frac{[complex\ ion]}{C_2\ [(S_2O_3)^{2-}]}$$

     Calculate the Ks for each complex ion solution and finds mean Ks

Result:-

Stability constant of the $Ag(S_2O_3^-)_2$ complex ion   =_____

<u>**Aim:-**</u>

Determine the standard free energy change ΔG° and equilibrium constant for the reaction potentiometrically.

<u>**Apparatus:-**</u>

Potentiometer assembly, Cu electrode, Ag electrode

<u>**Chemicals :-**</u>

0.1 M $CuSO_4$ solution, 0.1 M $AgNO_3$ solution, KNO_3 salt bridge.

<u>**Procedure:-**</u>

1) Prepare 0.05, 0.01 M $CuSO_4$ solution from 0.1M $CuSO_4$ solution.

2) Prepare 0.05, 0.01 M $AgNO_3$ solution from 0.1M $AgNO_3$ solution

3) Take 50 ml of .01 M $CuSO_4$ in a clean 100 ml beaker.

   Immerse Cu electrode in it and connect it to –ve terminal of the potentiometer.

4) Take 50 ml of 0.1 M $AgNO_3$ in a clean 100 ml beaker. Immerse Ag electrode in it and connect it to +ve terminal of the potentiometer.

5) Connect the two half cell by using KNO_3 salt bridge.

6) Construct the cell as.

   $^{(-)}$ Cu / $CuSO_4$ (0.01 M) // $AgNO_3$ (0.01 M) / $Ag^{(+)}$
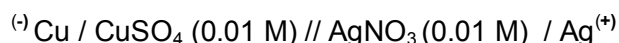
7) Note down the EMF of the cell in volt

8) Repeat the expt. by changing the concentration of $CuSO_4$ solution and $AgNO_3$ solution and record the EMF value for it.

Observation table:-

Sr.No.	CuSO$_4$ Conc.(m$_1$)	Activity Coeff.of CuSO$_4$ (γ_1)	AgNO$_3$ Conc.(m$_2$)	Activity Coeff. Of AgNO$_3$ (γ_2)	E$_{cell}$	E$^o_{cell}$
1	0.1	0.15	0.1	0.733		
2	0.05	0.31	0.05	0.812		
3	0.01	0.41	0.01	0.892		
				Mean		

Calculations:-

i) E^0,(Standard emf of the cell)

Using Nernst equation, we can write

$E_{cell} = E^o_{cell} - \log[Cu^{2+}][Ag]^2 / [Ag^+]^2 [Cu]$

By convention [Cu] = [Ag]=1 and

$[Cu^{2+}] = m_1\gamma_1$ and $[Ag^+] = m_2\gamma_2$

Substituting these in the above equation, we get

$E_{cell} = E^o_{cell} - \log[m_1\gamma_1 / [m_2\gamma_2]^2$

$E_{cell} = E^o_{cell} - \log m_1\gamma_1 / m_2^2\gamma_2^2$

Calculate E^0_{cell} and hence mean E^0_{cell}

ii) ΔG^0 (standard free energy change)

$\Delta G^0 = - n\,f\,E^0_{cell}$

i.e. $\Delta G^0 = -2 \times 96,500 \times E^0_{cell(mean)}$

$\Delta G^0 = $ ————————joule.

iii) K(equilibrium constant)

$E^o_{cell} = 0.0592 / 2 \times \log_{10}K$

$\log_{10}K = E^o_{cell} \times 2 / 0.0592$

i.e. $K = \text{Antilog}(E^o_{cell} \times 2 / 0.0592)$

Result:-

1) Mean E^o_{cell} =--------------volts.
2) ΔG^o for the reaction = _____joules.
3) Equilibrium constant K for the reaction = _____

pH –metry

Aim:-

Determine the Hammett constant of a given substituted benzoic acid by pH measurements.

Chemicals:-

0.01M benzoic acid, 0.02 M p-nitrobenzoic acid in 50% alcohol –water mixture , 0.025 M NaOH.

Procedure:-

1) Pipette out 50ml of benzoic acid solution in a beaker.

2) Measure its pH.

3) Add 0.2 ml of NaOH from the microburette, stir and measure the pH of the solution.

4) Repeat (3) until the equivalence point has been passed by one ml.

5) Repeat (1) to (4) for p-nitrobenzoic acid.

6) Present your observations as follows:

No.	ml of NaOH added (V)	pH	ΔpH	ΔV	ΔpH/ΔV	Mean V
1	0.0					
2	0.2					
3	0.4					
4	0.6					
.	.					
.	.					
.	.					
.	.					

7) Plot (i) pH Vs ml of NaOH.

   (ii) ΔpH/ΔV Vs mean V.

8) Ask for the value of reaction constant.

9) Calculate substituent constant.

Calculations: -

From the graph, determine the dissociation constant of benzoic acid (pK^o) and that of substituted benzoic acid (pK) from the value of pH at half neutralization points.

Then calculate Hammett constant'σ' by using the equation.

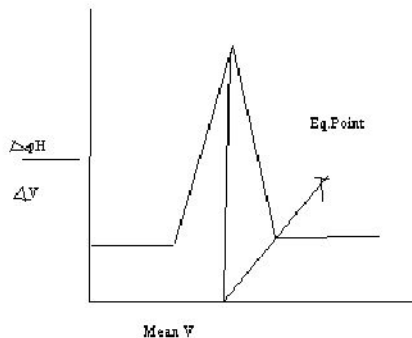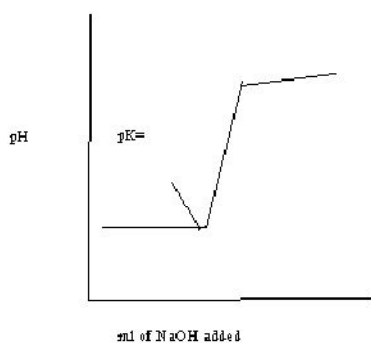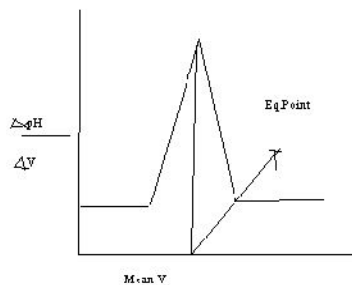$pK^o - pK = \rho \times \sigma$

Where,

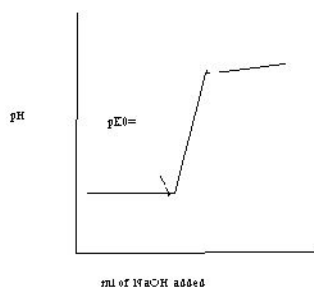σ = 1.65 for 50 % alcohol.

σ = pK° –pK / ρ   in aqueous solution.

p-nitro benzoic acid V: NaOH

benzoic acid V: NaOH

Result:-

1) Calculate substituent constant. (Hammett constant)=------------------

Aim :

Determine the amount of aspirin in the given tablet by pH metry.

Chemicals: Aspirin tablet, 0.1 M alcoholic KOH, 95% ethanol

Procedure:

1. Standardize the pH meter.

2. Grind the aspirin tablet to fine powder. Weigh it accurately.

3. Prepare 100 ml of aspirin solution in 100 ml volumetric flask using 95% ehanol.

4. Pipette out 25ml of aspirin solution in a beaker. Measure its pH.

5. Add 0.2 ml of KOH solution from the micro burette, stir and measure the pH of the solution.

6. Continue the addition of KOH solution until the equivalence point has been passed by one ml.

   Present your observations as follows

No.	ml of KOH added (V)	pH	ΔpH	ΔV	$\Delta pH/\Delta V$	Mean V
1	0.0					
2	0.2					
3	0.4					
4	0.6					
.	..					
.	..					

7. Plot (i) pH Vs ml of KOH added.

   (ii) $\Delta pH/\Delta V$ Vs Mean V

8. Find equivalence point V_1 from the graph. Calculate the amount of aspirin in the given ta1blet using appropriate relation.

Calculations :

Calculation of quantity of aspirin in the given solution

1 moles of KOH $\underline{=}$ 1 mole of Aspirin $\underline{=}$ 180 gm of Aspirin

    1000 ml of 1 M KOH $\underline{=}$ 180 gm of Aspirin

    V_1 ml of 0.1 M KOH $\underline{=}$ $\dfrac{V_1 \times 180 \times 0.1}{1000}$

                           = $0.018 \times V_1$ gm of Aspirin

25 ml of aspirin solution $=$ $0.018 \times V_1$ gm of Aspirin

100 ml of aspirin solution $=$ $0.018 \times 4 V_1$ gm of Aspirin

                           = A gm of Aspirin

W gm of drug sample $=$ A gm of sample

 100 gm of drug sample $=$ $\dfrac{A \times 100}{W}$ % of sample

Result:

Amount of aspirin in the given tablet =---------------------------------------

Colorimetry/ Spectrophotometry

Aim:-

To determine the pK_a and K_a values of acid base indicator colorimetrically

Chemicals:-

0.05 % indicator solution. 10 buffer solutions of unknown pH, buffer solution of pH 4.

Procedure:-

1) Measure the pH of all the buffer solutions using pH-meter.
2) Take appropriate volume (0.2 ml or 0.3 ml) of the indicator solution by means of the graduated pipette into a clean cuvette. Pipette out of 10ml of the buffer solution no.1 into it. Shake well and measure the absorbance.
3) Repeat (2) for all the remaining buffer solutions.
4) Present your observations as follows:

   λmax = _____

Buffer solution no.	pH	Absorbance A	A_1-A A-A_2	$\log \left(\dfrac{A1 - A}{A - A2} \right)$
1(of minimum pH)				
2				
3				
4				
5				
6				
7				
8				
9				
10(of maximum pH)				

5) Plot (i) $\log \left(\dfrac{A1-A}{A-A2} \right)$ Vs pH
       (ii) A Vs pH

   Calculate pKa and hence Ka from graph and calculations.

Result:-

1) pK_a from calculations   = _____

2) K_a from calculations   =_____

3) pK_a from graph   =_____

4) K_a from graph   =_____

reparationS

1% methyl red indicator in 50% ethyl alcohol , 0.1M NaOH

Ten buffer soln from pH 3 to 8

Preparation of ten buffer soln by using 0.2M $Na_2HPO_4 .2H_2O$ and 0.1 M citric acid,

Methyl indicator = 0.05gm Methyl powder to 100 ml distilled water

Sr. No.	X ml of (A) Na_2HPO_4	V ml of (B) citric acid	pH of solution
1	20.55ml	79.45 ml	3.0
2	38.55 ml	61.45 ml	4.0
3	46.75 ml	53.75 ml	4.6
4	57.50 ml	47.50 ml	5.0
5	58.00 ml	42.00 ml	5.6
6	63.15 ml	36.85 ml	6.0
7	72.15 ml	27.25 ml	6.6
8	82.35 ml	17.65 ml	7.0
9	93.65 ml	6.35 ml	7.6
10	97.25 ml	2.75 ml	8.0

Graph
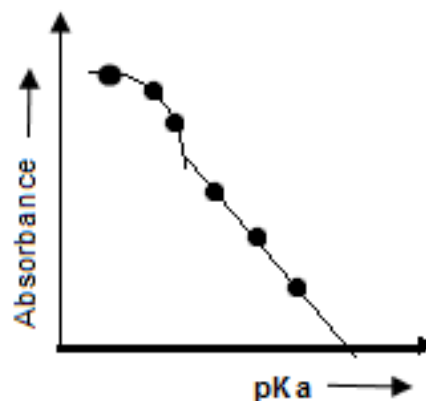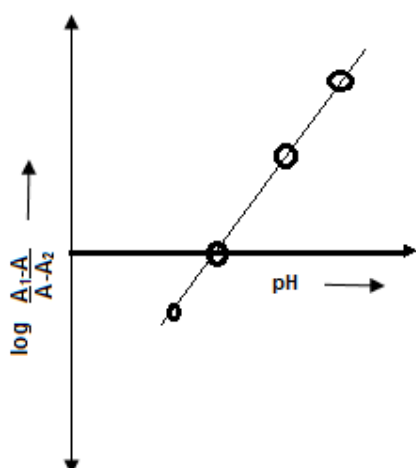
<u>**Aim:-**</u>

Determine the empirical formula of Ferric -5-sulpho Salicylate complex by Job's method and verify by slope ratio method.

<u>**Chemicals :-**</u>

0.001N HCl solution, 0.001N 5-sulpho Salicylic acid in 0.001 N HCl solution,

0.001 N Ferric nitrate solution,

<u>**Procedure:-**</u>

1) Prepare the following mixtures using 0.001 M Ferric nitrate and 0.001N 5-sulpho Salicylic acid solution.

Fe^{3+} sol. in ml	1	2	3	4	5	6	7	8	9
5-SA sol. in ml	9	8	7	6	5	4	3	2	1

2) Using 5+5 mixture determine λ_{max}
3) Note down O.D. of each solution at λ_{max}
4) For the slope ratio method prepare following two set of mixture solution.

Set- I

Fe^{3+} sol. in ml	10	10	10	10	10
5-SA sol. in ml	1	2	3	4	5
0.001N HCl	39	38	37	36	35

Flask No.	Absorbance
1 2 3 . .	

Set- II

Fe^{3+} sol. in ml	1	2	3	4	5
5-SA sol. in ml	10	10	10	10	10
0.001N HCl	39	38	37	36	35

Note down O.D. of each solution

Flask No.	Absorbance/O.D.
1 2 3 . .	

Calculations:-

For Job`s method:

Plot a graph of O.D. Vs Composition

The maxima of curve shows the proportion of metal ion & SA in the complex formation
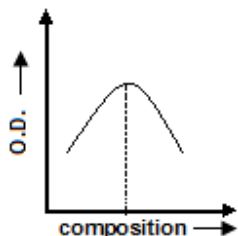
For slope ratio method:

For set-I plot the graph of O.D. Vs volume of 5-SSA added. Find out slope of straight line =Slope I

For II Slope: Plot O.D. Vs volume of Fe^{3+} ion solution added. Find out the slope of
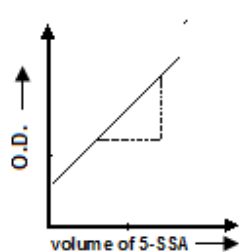
straight line = Slope II
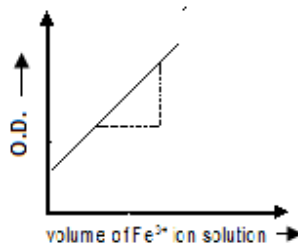
The ratio Slope I/ Slope II gives empirical formula

Explanation about the relation between slope ratio and Empirical formula

From the ratio of slope we gwt the idea about metal to ligand complex ratio

If the slope ratio is one then Empirical formula M:L => 1:1

Result:- Empirical formula of complex =_____

Surface Chemistry

Aim :-

To Determine the Critical Micelle Concentration of Potassium laurate/ Sodium laurate by surface tension method

Apparatus: Stalgmometer, beaker volumetric flask, burette pipette, sp. Gravity bottle

Chemicals:- Distilled water, 0.2 M Potassium laurate solution Stock solution
　　　　　　 0.1M, 0.05M, 0.025M. 0.0125M solution of Potassium laurate

Procedure:

A) Preparation of solution

1) Prepare the 0.2M solution of potassium Laurate (mol.wt=-238.4) in Distilled water
2) Then by successive dilution of stock solution prepare 0.1M, 0.05M., 0.025 M
0.0125M solution of Potassium Laurate.

B) To determine density of solution:

1) Take sp. gravity bottle / Pyknometer with distilled water and acetone and dry it
2) Take the weight of sp. gravity bottle along with stopper
3) Fill the bottle with distilled water \and insert the stopper in it. Remove all water drops from surface of bottle and weigh it
4) Empty the bottle and dry it again
5) Similarly take Various concentration of potassium Laurate in bottle and weigh it.

C) To determine number of Drops:

6) Clean the Stalgmometer with distilled water and acetone and dry it.
7) Fill the Stalgmometer with distilled water and determine number of drops form the fixed volume of water between two marks.
8) Rinse the Stalagmometer by 0.02M solution of potassium Laurate
9) Fill the Stalgmometer by 0.02M solution of potassium Laurate determine number if the drops formed from the fixed volume of solution between the two marks
10) Repeat the procedure 3to4 for 0.1M, 0.05M, 0.025M. 0.0125M solution

Observations:

1. Room Temperature = ------------^{o}C
2. Wt of empty sp. gravity bottle/ Pyknometer = -------------------gm
3. Density of Water =------------------g/cm^{3}
4. Surface Tension of Water = 7.169 x 10 N/m

Observation Table:

A) To Determine the density of solution.

Concentration	Wt of empty sp. Bottle+ Wt of liquid	Wt of liquid	Density= W_l/W_w
Water		W_W	$D_W = 1$
0.2M			
0.1M			
0.05M			
0.025M			
0.0125M			

B) To Determine the number of drops

Concentration C	logC	Density	Number of drops	Surface Tension γ
Water				
0.2M				
0.1M				
0.05M				
0.025M				
0.0125M				

Calculatios :

A) To Determine the density of solution.

$$\text{Density} = \frac{\text{Wt of liquid}}{\text{Wt of water}}$$

B) To determine the Surface Tension of solution:

$$\gamma_{liquid} = \frac{\text{Number of drops of Liquid (nl) X Density of Liquid (Dl)}}{\text{Number of drops of water (nw) X Density of water (Dw)}} \times \gamma_{water}$$

Graph: Plot the graph of γ against logC

$logC = X$

$C = Antilog\ X$
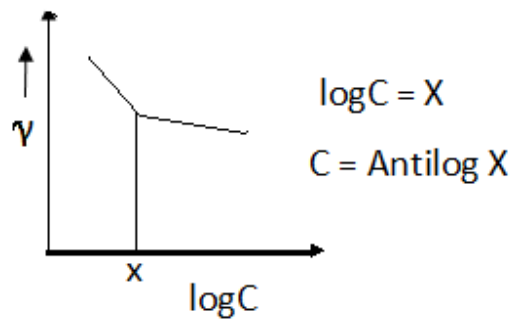
Result :

The critical Micelle concentration (CMC) of potassium Laurate (C)--------

Aim:-

Determine Critical Micellar Concentration (CMC) of soap by surface tension method

Chemicals:- 0.2 M potassium laurate (soap)

Procedure:-

1) Prepare 0.1, 0.075, 0.05, 0.025, 0.02, 0.01, 0.005, 0.0025 and 0.001M solutions of   potassium laurate from the stock solution.
2) Determine number of drops for distilled water and each of the above solutions between two marks of stalagmometer.
3) Determine density of each solution using pyknometer / specific gravity bottle.
4) Calculate Surface tension of each liquid using appropriate relation.

Present your observations as follows

Concentration C (M)	Number of drops (n)	Density (d)	Surface tension
Distilled Water	n_w	Dw	Sw(given)
0.1			
0.075			
0.05			
0.025			
0.02			
0.01			
0.005			
0.0025			
0.001			

5) Plot a graph of surface tension Vs concentration of potassium laurate
6) Determine Critical Micellar Concentration from the graph.

Calculatios :

C) To Determine the density of solution.

$$\text{Density} = \frac{\text{Wt of liquid}}{\text{Wt of water}}$$

D) To determine the Surface Tension of solution:

$$\gamma_{liquid} = \frac{\text{Number of drops of Liquid (nl) X Density of Liquid (Dl)}}{\text{Number of drops of water (nw) X Density of water (Dw)}} \times \gamma_{water}$$

Graph:
Plot the graph of γ against logC

logC = X

C = Antilog X
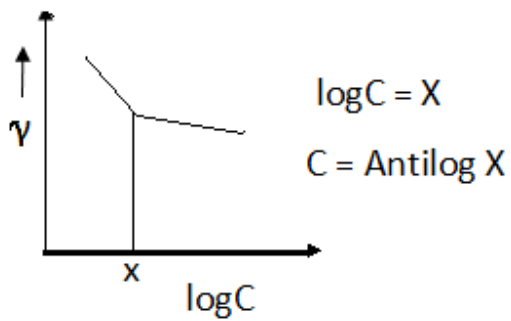
Result : The critical Micelle concentration (CMC) of Soap(C)---------------
7)

.

NON INSTRUMENTAL

Chemical Kinetics

Aim:

To determine the rate constant for the depolymerisation of diacetone alcohol catalysed by sodium hydroxide using dilatometer.

Apparatus:

Dilatometer, 50 ml burette. 5 ml graduated pipette, 100 ml stoppered conical flask (or beaker,) stopwatch

Chemicals:

Redistilled diacetone alcohol, 0.4 M NaOH

Prodcedure:

(1) Take the tap out and remove as much grease as possible from it and form the cell with tissue and carbon tetrachloride.Then clean the cell and the tap with chromic-suplhuric acid cleaning mixture. Rinse thoroughly several times with distilled water. Allow the water to drain and then rinse two or three times with small quantities of dry acetone. Dry the dilatometer by blowing warm air through it. Then tap should then be carefully lubricated with low melting 19 vaccume grease.

(2) Fasten the dilatometer in a water bath so that all the bulb is immersed.

(3) Take exactly 50 ml of 0.4M NaOH in the 100 ml stopnered conical ilask and leave it along with the bottle of diacetone alcohol in the water bath for about 10 to 15 minutes.

(4) Transfer 3 ml of diacetone alcohol into the conical flask containing NaOH and mix well.

(5) Carefully, pour the solution into the dilatometer.

(6) Open the tap and adjust the level of the meniscus in the capillary until it stands near the bottom of the scale.

(7) Then close the tap. The dilatometer must, of course, be free from air bubbles.

(8) Start the stop watch when the meniscus of the liquid reaches a graduation mark on the capillary.

(9) Take the readings of the position of the meniscus every minute for a period of 10 minutes.

(10) Take a second set of readings after a fixed interval of 10 minutes.

Observation Tables

Time t (min)	Reading r	Time (t+T) (min)	Reading r'	r'-r	$\log_{10}(r'-r)$

Graph:

Plot $\log_{10}(r'-r)$ Vs t

Calculations:

Calculate the velocity constant from the slope.

$$Slope = \frac{k}{2.303}$$

Result:

Velocity constant of the reaction = --

<u>**Aim:-**</u>

To determine rate constant of the reaction between potassium persulphate and KI and study the influence of ionic strength.

Chemicals: 0.05 N $K_2S_2O_8$, 0.05 N KI, 0.4 N KCl, 0.005 N $Na_2S_2O_3$,

<u>**Procedure:-**</u>

1) Prepare the following mixtures in 250 ml stoppered bottles

Bottle no	ml of $K_2S_2O_8$	ml of KCl	ml of Water
1	20	00	60
2	20	20	40
3	20	40	20
4	20	60	00

2) Place these bottles and the bottle containing 20 ml KI solution in water bath for 10 minutes
3) Add KI solution in the Bottle No 1 and start the stop watch.
4) Titrate 10ml of the reaction mixture in presence of ice against Sodium thiosulphate solution using starch as an indicator. at the intervals of 5, 10, 15, 20, 25 and 30 minutes from the start.
5) Repeat (3) and (4) for the remaining bottles.
Present your observations as follows

Set No: --

Time (min)	Titre reading (x) ml	a-x	$\dfrac{x}{a-x}$
5			
10			
15			
20			
25			
30			

6) Prepare similar observation table for all the sets.

Set No	K	log $_{10}$ k	μ	μ$^{1/2}$
1			0.04	
2			0.12	
3			0.20	
4			0.28	

7) Plot i) $\frac{x}{a-x}$ Vs t for each set   ii) log k Vs μ$^{1/2}$..

8) Calculate initial concentration (a). Calculate k for each set from graph (i). Comment on the influence of ionic strength using graph (ii).

<u>**Aim:-**</u>

Determine the order of reaction between potassium persulphate & KI by fractional change method

Chemicals :

0.05 N $K_2S_2O_8$, 0.05 N KI, 0.005 N $Na_2S_2O_3$, Starch indicator

<u>**Procedure:-**</u>

1) Take 50ml of Potassium persulphate solution & 50 ml KI solution in two dry bottles separately.

2) Keep both the bottles in water bath to attain the room temperature. Mix the two solutions & note the time of mixing.

3) Titrate 10ml of the reaction mixture against Sodium thiosulphate solution at the intervals of 5, 10, 15, 20, 25 and 30 minutes from the start.

4) Repeat (1) to (3) by mixing 25ml of each solution and 50ml distilled water.

5) Calculate the initial concentration of reactants in each case.

6) Present your observation as follows:

Set I: a_1 = -----   Set II: a_2 = -------

Time in minutes	Set I		Set II	
	Titre reading	X	Titre reading	X
5				
10				
15				
20				
25				
30				

7) Plot X Vs time for each set. Hence, find the time required to complete half the reaction in each case.

8) Calculate the order of reaction

<u>**Result:-**</u>

Order of reaction (fractional change method)=   _____

NON INSTRUMENTAL

Aim:-

To determine the transport number of H^+ and Cl^- ions in HCl by moving boundary method.

Apparatus : .

1 ml glass capillary tube with 0.01 ml graduations fitted with Cu electrode (anode) at the bottom Ag-AgCl electrodes, D.C. power supply, stop-watch. 50 ml beaker miliammter, rheostat.

Chemicals :-

0.1 M HCl methyl orange indicator (solid)

Procedure:-

(1) Take about 25 ml of 0.1 M HCl in a 50 ml beaker

(2) Add to it sufficient solid methyl orange to give it enough pink-red colour to be seen in the capillary.

(3) Clean the capillary with chromic—sulphuric acid mixture. wash with water and then rinse with the coloured HCl solution.

(4) Fill the capillary with HCl solution taking care that no air bubble is trapped in the capillary.

(5) Insert the Ag-AgCl electrode (cathode) at the top.

(6) Set up the whole apparatus in a glass sided tank of water to prevent disturbances of the boundary' by temperature change.

(7) Connect the electrodes to D.C. power supply through a milliameter and rheostat in ries and switch on the current at once. A current of 10 mA is convenient to form the boundary and bring it into the calibrated volume rapidly. It may then be reduced to a constant value between 3 to 5 mA.

(8) A very sharp boundary-red above, yellow below can be seen best by placing a thin sheet of white paper behind the apparatus and an electric light bulb behind the paper.

(9) As soon as the boundary reaches the first graduation on the capillary, start the stop-clock.

(10) Note the time (without stopping the clock) when the boundary reaches each graduation up to the top or the tube.

Observation table

Volume Swept V ml	Total time t (sec)

Graph :

Plot volume Vs time $t_H{}^+$

Calculations :

Find the slope of the straight line graph passing through origin.

Calculate the transport number of H ion using the relation,

$$tH+ = \frac{CF}{1000I} \times \frac{dv}{dt}$$

C = 0.1 M,   F = 96500 Coulombs

I = current in ampere

$tCl^- = 1 - tH^+$

Results :

1) Transport no. of H⁺=_____

ii) Transport no. of Cl⁻ =_____

SEM -II

INSTRUMENTAL:-

Conductometry:-

1) Determination of the dissociation constant of acetic acid in DMSO, DMF, acetone and dioxane by titrating it with KOH.

2) Determination of critical micellar concentration (CMC) of sodium lauryl sulphate from the measurement of conductivities at different concentrations.

Potentiometry:-

1) To determine the amount of each halide in a mixture of halides containing a) KI and KBr/KCl or b) KI, KBr and KCl potentiometrically.

2) To titrate ferrous ammonium sulphate solution with potassium dichromate solution potentiometrically using bimetallic electrode pair.
.

pH -metry:-

1) To determine acidic and basic dissociation constants of an amino acid and hence the isoelectric point of the acid.

2) To determine the three dissociation constants of polybasic acid such as H_3PO_4 by pH measurements.

Colorimetry/ Spectrophotometry:-

1) Determination of iron in water using a colorimeter.

2) Simultaneous determination of $Cr_2O_7^{2-}$ and MnO_4^- ions or Co^{2+} and Ni^{2+} in the solution by spectrophotometry.

3) Record the UV spectrum of Benzene, Pyridine and Pyrimidine in methanol. Compare and discuss the various transition involved in terms of MO theory.

Polarimetry:-

1) Polarimetric determination of the specific rotation of camphor in benzene and carbon tetrachloride.

2) Determine the percentage of two optically active substances (d-glucose and d-tartaric acid) in a mixture polarimetrically.

NON INSTRUMENTAL:-

Chemical kinetics:-

1) To investigate the kinetics of iodination of acetone.

2) To determine energy of activation of the hydrolysis of methyl acetate in presence of hydrochloric acid (Calculations and graphs expected from excel programming).

Non instrumental:-

1) To obtain solubility curve for liquid say water-acetic acid-chloroform system.

2) Determination of partial molar volume of ethanol in dilute aqueous solutions.

3) To study the effect of addition of an electrolyte (KCl, NaCl, NH_4Cl, Na_2SO_4, K_2SO_4) on solubility of an organic acid(benzoic acid or salicylic acid).

Conductometry

Aim:- Determine Critical Micellar Concentration (CMC) of sodium lauryl sulphate[Soap soln] from the measurement of conductivities at different concentrations

Chemicals:- 0.02 M KCl, 0.1 M Sodium lauryl sulphate

Procedure:-

1) Prepare 2 and 1×10^{-2} M, 9,8,7,6,5,4,3,2,and 1×10^{-3} M solutions of sodium lauryl sulphate.

2) Measure the conductance of 0.02 M KCl. Ask for specific conductance and calculate cell constant.

3) Measure the conductance of each of the solution of sodium lauryl sulphate. Calculate specific conductance and equivalent conductance.
   Present your observations as follows

Concentration C (M)	Conductance Mho	Specific Conductance (k)	Equivalent Conductance (Λ)	$C^{1/2}$
2×10^{-2}				
1×10^{-2}				
9×10^{-3}				
8×10^{-3}				
7×10^{-3}				
6×10^{-3}				
5×10^{-3}				
4×10^{-3}				
3×10^{-3}				
2×10^{-3}				
1×10^{-3}				

4) Plot a graph of i) Specific conductance Vs Concentration
   ii) Equivalent conductance Vs $C^{1/2}$

5) Determine Critical Micellar Concentration from the graph.

SLS in Water

Observed Conductance

Concentration

Equivalent conductance

$\sqrt{c}$

SLS in Water

Observed Conductance

Concentration

Equivalent conductance

$\sqrt{c}$
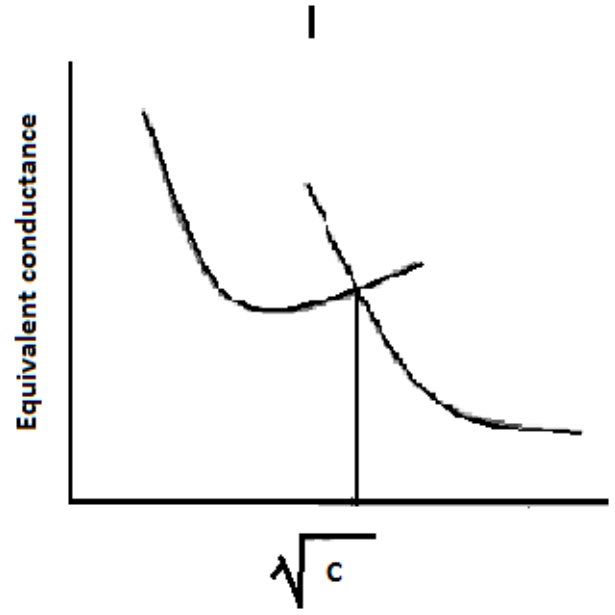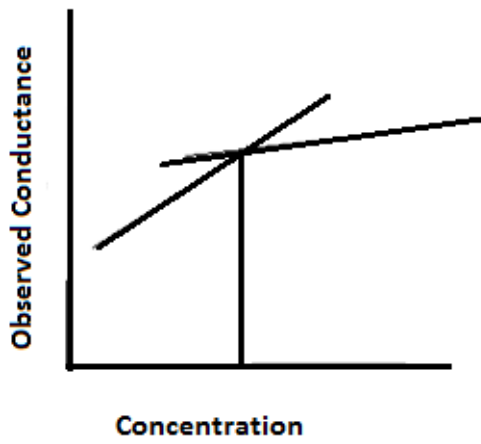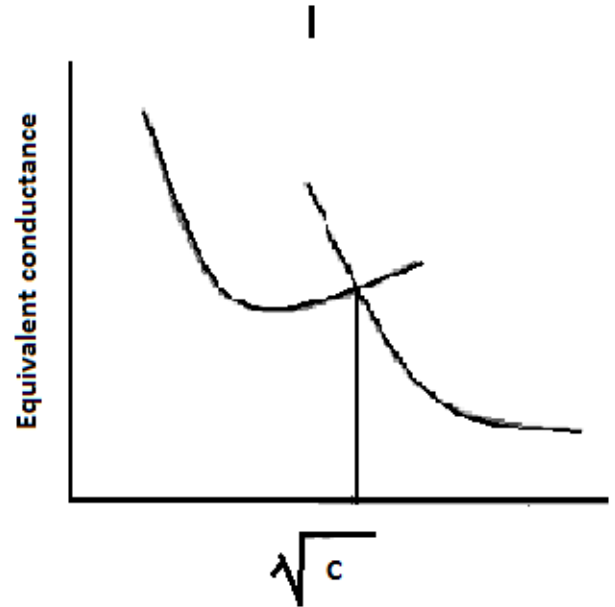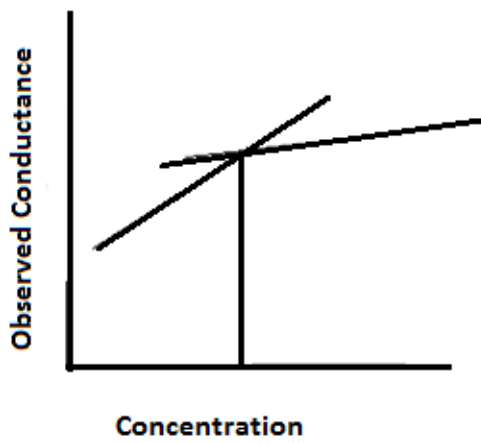
Result

Critical Micellar Concentration (CMC) of

sodium lauryl sulphate[Soap soln]   = ...

Potentiometry

Aim:- To determine the amount of each halide in a binary mixture of halides

containing (a) KI and KCl or (b)KI and KBr potentiometrically.

Apparatus:- Potentiometer assembly , two Ag electrodes , KNO3/NH4NO3 –agar

bridge , two 50 ml beakers ,10 ml pipette , 10 ml microburrete.

Chemicals:-0.01M $AgNO_3$, and mixture of halides.

Procedure:-

1) Standardize the potentiometer.
2) Pipette out 10 ml of given mixture of halides in 50 ml of beaker. Add to it about 10 ml of distilled water and dip in it a clean polished Ag electrode.
3) Pipette out 10 ml of 0.01 M silver nitrate solution in another 50 ml beaker .Add to it about 10 ml of distilled water and dip in it a clean polished Ag electrode .
4) Connect the two half cells by the salt bridge to form the cell,
   Ag / Halide mixture // 0.01 M $AgNO_3$ Solution / Ag.
5) Connect the cell to potentiometer and measure its emf.
6) Fill the micro burette with 0.01 M AgNO3 Solution. Add 0.5 ml of silver nitrate solution to the Halide mixture , stir on magnetic stirrer and measure the emf of the cell.
7) Continue the addition of 0.5 ml of the silver nitrate solution to the Halide mixture and measurement of the emf of the cell each time until two sharp falls in emf are obtained

Observation table:-

No.	ml of $AgNO_3$ added	EMF(E)	ΔE	ΔV	ΔE/ΔV	Mean volume of $AgNO_3$ ml
1	0.0					
2	0.5					
3	1.0					
4	1.5					
.	.					
.	.					
.	.					
.	.					
.	.					
.	.					
.	.					
.	.					

Graph :-

Plot 1) emf Vs ml of silver nitrate solution added.

2) $\Delta E / \Delta V$ Vs mean volume of silver nitrate solution .

Calculation :-

Find the volumes X_1 and X_2 of 0.01 M $AgNO_3$ Solution corresponding to the two maxima .

Equivalent Point of KI = X_1 ml.

Equivalent Point of KCl /KBr = (X_2-X_1) ml.

Calculate the molarity and g/l of KI and KCl / KBr

M_2 = 0.001 x X_1 and g/l = M_2 x 166

For KCl

M_2 = 0.001 x (X_2- X_1) and g/l = M_2 x 74.5

For KBr

M_2 = 0.001 x (X_2- X_1) and g/l = M_2 x 118

Result:-

1. Equivalence point of KI   =_____ N
2. Strength of KI in a mixture   = _____ gm/lit.
3. Equivalence point of KCl /KBr   =_____ N
4. Strength of KCl /KBr in a mixture = _____ gm/lit

Preparation of solutions

1. 0.01 M $AgNO_3$ solution : 1.7 g/l
2. 0.1 M $Na_2S_2O_3$ solution : 24.8 g/l
3. 0.01 M KI solution : 1.66 g/l
4. 0.01 M KBr solution : 1.18 g/l
5. 0.01 M KCl solution : 7.45 g/l
6. Following mixtures of halides may be given for titration.

0.01 M KI ml	0.01 M KCl ml / KBr ml
40	60
50	50
60	40

7. Preparation of the salt [bridge]

Dissolve about 3 g of the salt and 0.3 g of agar powder in 10 c.c. of water. Heat in a small beaker on a steam- bath. when a clear solution is obtained , it is poure while hot into an U-tube made form glass tubing. Great care must be taken to avoid air bubbles in the bridge which will break the circuit. The mixture sets to a gel on cooling.

Literature values :-
Stability constant of $Ag(S_2O_3)$ complex ion = 1×10^{13}

Aim:-

Titrate ferrous ammonium sulphate solution with potassium dichromate solution potentiometrically using bimetallic electrode pair

Chemicals: 0.1 N $K_2Cr_2O_7$, 0.1 N (approx) ferrous ammonium sulphate in 2N H_2SO_4, 2N H_2SO_4

Procedure:-

1) Standardize the potentiometer.

2) Take 10ml of ferrous ammonium sulphate solution in a 50 ml beaker. Add to it 10 ml of 2N H_2SO_4.

3) Fill the burette with 0.1 N $K_2Cr_2O_7$.

4) Clean the tungsten electrode(W) either by polishing with emery paper or by immersing into just molten sodium nitrite for a few seconds and then washing with distilled water.

5) Set up the following cell

   –W | ferrous ammonium sulphate | Pt+

   Measure emf of the cell.

6) Add 0.5 ml $K_2Cr_2O_7$ from burette to the ferrous ammonium sulphate solution Stir and measure emf of the cell.

7) Repeat (6) till the equivalence point exceeds by 2 ml.

   Present your observations as follows.

No.	ml of $K_2Cr_2O_7$ solution added (V)	EMF (E)	ΔE	ΔV	$\Delta E/\Delta V$	Mean V
1	0.0					
2	0.5					
3	1.0					
4	1.5					
5	2.0					
..	..					
..	..					

8)Plot   (i) EMF Vs ml of $K_2Cr_2O_7$ solution added.

(ii)$\Delta E / \Delta V$ Vs mean volume of $K_2Cr_2O_7$ solution

9) Find out equivalence point from the graph and calculate normality and   strength of ferrous ammonium sulphate solution.
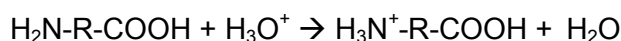
.

pH -metry

Aim:-

To determine acidic and basic dissociation constant of an amino acid and hence iso –
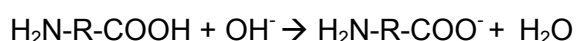electric point of the acid.

Theory

Amino acid are ampholytes i.e. they have separate acidic and basic groups.

They can be generally represented as H_2N-R-COOH. Addition of acid results in
neutralization of –NH2 group.

H_2N-R-COOH + H_3O^+ → H_3N^+-R-COOH + H_2O

Addition of strong base gives

H_2N-R-COOH + OH^- → H_2N-R-COO⁻ + H_2O

These can be determined by simplified technique where an amino acid is considered as
dibasic acid. Consider the hydrochloride of glycine:
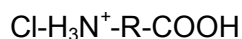
Cl-H_3N^+-R-COOH

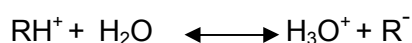When this is neutralized by alkali there are two stages of reaction corresponding to the two
stages of neutralization of dibasic acid:

H_3N^+-R-COOH + OH^- → H_3N^+-R-COO⁻ + H_2O

H_3N^+-R-COO⁻ + OH^- → H_2N-R-COO⁻ + H_2O
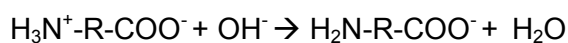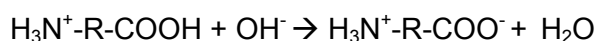
Replacing H_3N^+-R-COO⁻ by RH+ H_3N^+-R-COOH, by RH_2^+ and H_2N-R-COO⁻ by R⁻

 We get

  RH_2^+ + H_2O ⟷ H_3O^+ + RH^+

  RH^+ + H_2O ⟷ H_3O^+ + R⁻

The position of equibrillium varies woth the nature of amino acid and the pH of the solution.
In strongly acidic solution the acid is mainly in cationic form whereas in strongly basic
solution at which the concentration of the dipolar ion is maximum, is known as isoelectric
point usually denoted by pI of amino acid.

Isoelectric point (pI) of amino acid

$$pI = \frac{pK1 + pK2}{2}$$

Chemical: –0.1 N of amino acid (glycine) solution, 0.1 N NaOH solution , 0.1 N HCl
solution, Formaldehyde

Procedure: – Standardize the pH meter.

Part-I

1. Pipette out 10 ml of 0.1 N amino acid solution in 100 ml beaker. Add 40 ml distilled water. Add 5 ml of formaldehyde in the solution.
2. Fill burette with 0.1 N NaOH solution.
3. Insert the electrode in the glycine solution & record initial pH
4. Add 0.5 ml of NaOH solution to glycine solution and stir and measure pH of the solution
5. Repeat (4) until the equivalence point has been passed by 2 ml.

Part-II

Similarly for titrating amino acid with HCl solution, pipette out 10 ml of 0.1 N amino acid solution in a beaker. Add 40 ml distilled water and titrate against 0.1 N HCl solution to decrease pH upto 1.5.The sharp decrease in pH shows the equivalence point.
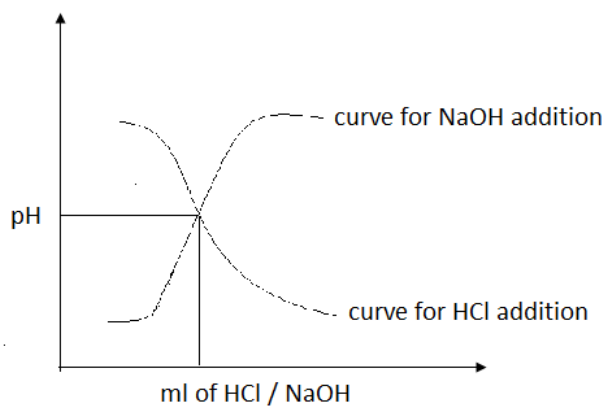
Present your observations as follows.

Part I

No.	ml of NaOH added (V)	pH	ΔpH	ΔV	ΔpH/ΔV	Mean V
1	0.0					
2	0.5					
3	1.0					
4	1.5					
.	.					
.	.					
.	.					

Prepare similar table for Part-II

6. Plot (i) pH Vs ml of NaOH

(ii) $\Delta pH/\Delta V$ Vs mean V.
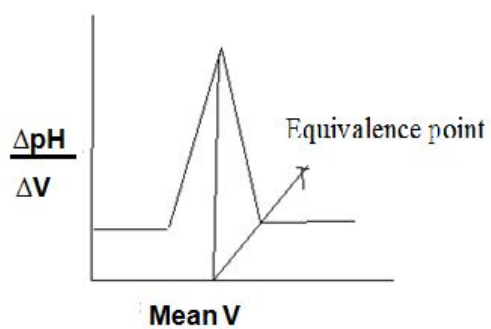
7. Find pk_1 and pk_2 values and calculate iso-electric point of the amino acid.

Results:-

1. pk_1 of amino acid =
2. pk_2 of amino acid =
3. Isoelectric point of amino acid =

Aim:- Determine the three dissociation constants of polybasic acid such as H_3PO_4 by pH measurements.

Apparatus :-

pHmeter, glass electrode, sat calomel electrode, magnetic stirrer, 50 ml beaker, 10 ml microburette,25 ml pipette

Chemicals:-

0.05 M Phosphoric acid , 0.5 M NaOH , 0.05 M Potassium hydrogen phthalate (pH =4) ,$CaCl_2$

Procedure:-

1) Take about 25 ml of potassium hydrogen phthalate solution in the 50 ml beaker. Dip the glass electrode and the saturated calomel electrode into it. Connect the electrodes to pH- meter. Adjust the scale to pH 4.The pH—meter is standardized

2) Wash the electrodes and the beaker with distilled water. Pipette out 25 ml of the phosphoric acid solution in the beaker. Dip the electrodes into it and mpasure the pH

3) Fill the microburette with NaOH solution

4) Add 0.2 ml of NaDH solution to the phosphoric acid solution Stir and measure the pH.

5) Continue the addition of NaOH from the burette,stir and measure the pH after the addition of each 0.2 ml till the pH of the solution is nearly 10.

No.	ml of NaOH added (V)	pH	ΔpH	ΔV	ΔpH/ΔV	Mean V
1	0.0					
2	0.2					
3	0.4					
4	0.6					
.	.					
.	.					

Graph

Plot (i) pH Vs ml of NaOH added.

(ii) $\Delta pH/ \Delta V$ Vs means V

Calculations:-

(1) Find the volumes X1 and X2 of 0.5 H NaOH corresponding to the two Maxima on the $\Delta pH/\Delta V$ Vs mean V graph.
First equivalence point = X1 ml
Second equivalence point = X3 ml
(2) The pH of the solution at the half neutralization points of first and second stages of neutralization are equal pKa_1 and pKa_2 respectively.

pH at ½ X1 = pKa_1

pH at ½ X2 = pKa_2

(3) Calculate Ka_1 & Ka_2

$$pKa_1 = -\log Ka_1$$
$$pKa_2 = -\log Ka_2$$

Result:-

1) First ionization constant of phosphoric acid   =_____

2) Second ionization constant of phosphoric acid =_____

3) Third ionization constant of phosphoric acid =_____

Preparation of solutions :

1] 0.05M Potassium hydrogen phthalate = 10.21 g/1

2] 0.05" H_3PO_4 = 4.9 g/1. Or 2:72 ml/l

Ionization constants of H_3PO_4

pKa_1 = **2.12**	$Ka_1 = 7.5 \times 10^{-3}$
pKa_2 = **7.21**	$Ka_2 = 6.2 \times 10^{-8}$

Colorimetry Spectrophotometry

Aim   : To Determine the concentration of iron in water using a colorimeter.

Chemicals   : 4.0 M HCl, 10 % Potassium thiocynate solution, 50 ppm iron solution.

Procedure :

1. Prepare standard solutions for calibration curve as follow

Flask No.	Con. in ppm	ml of 10 ppm iron solution	ml of 0.4 M HCl solution	ml of 10 % Potassium thiocynate solution
Blank	0.0(Blank)	0	5	5
1	1	1	5	5
2	2	2	5	5
3	3	3	5	5
4	5	5	5	5
5	7	7	5	5
6	8	8	5	5
7	10	10	5	5
8	Unknown	Unknown	5	5

Dilute the solution in each volumetric flask to 50 ml using distilled water.

2. Adjust zero absorbance in colorimeter using blank solution.
3. Using 2 ppm solution determine λ_{max} .
4. Measure the absorbance of each solution at λ_{max} .

Present your observations as follows

Flask No	Conc of Fe^{+2} (mg/l)	Absorbance
1	1	
2	2	
3	3	
4	5	
5	7	
6	8	
7	10	
----	Unknown	

1. Prepare calibration curve by plotting absorbance Vs Conc. of Fe^{+2} solution.
2. Find unknown concentration of Fe^{+2} in the given water sample using calibration curve.

Result :

unknown concentration of Fe^{+2} in the given water sample = ……………………

Preparation

50ppm Iron solution = weigh 175 mg of ferrous ammonium sulfate and dissolve in 100 ml of H2O and then add 1 mL of concentrated H2SO4. Dilute this solution in a clean 500 ml volumetric flask up to the mark and mix thoroughly.

Aim:-

Determine $Cr_2O_7^{2-}$ and MnO_4^- **or** Ni^{2+} and Co^{2+} concentrations simultaneously in the solution by spectrophotometry.

Chemicals:- 0.01 M $K_2Cr_2O_7$ and 0.01M $KMnO_4$ prepared in 1N H_2SO_4

or

0.2M $CoSo_4$ solution and 0.5M $NiSO_4$ solution , Mixture of unknown concentration.

Procedure:-

1) Prepare 0.001, 0.002,0.004, 0.006 and 0.008 M Solutions of $K_2Cr_2O_7$ and $KMnO_4$ in 1N H_2SO_4

or

Prepare 0.1 M, 0.2 M, 0.3M and 0.4 M Nickel Sulphate solutions and 0.04 M, 0.08M, 0.12 M, and 0.16 M Cobalt sulphate solutions.

2) Measure the absorbance of all the solutions including the mixture of unknown concentration at the wavelengths of λ_1 and λ_2 .

(λ_1 = 440 nm and λ_2 = 545 nm for $K_2Cr_2O_7$ and $KMnO_4$ system)

(λ_1 = 510 nm and λ_2 = 670 nm for $NiSO_4$ and $CoSo_4$ system)

3) Present your observations as follows:

Flask No.	Solution	Concentration (M)	Absorbance	
			At λ_1	At λ_2
1		0.001/ 0.1		
2		0.002/ 0.2		
3	$Cr_2O_7^2$ /Ni^{2+} solution	0.004/ 0.3		
4		0.006/ 0.4		
5		0.008/ 0.5		

6		0.001/ 0.04		
7		0.002/ 0.08		
8	MnO_4^-/Co^{2+} solution	0.004/ 0.12		
9		0.006/ 0.16		
10		0.008/ 0.20		
11	Mixture of unknown conc.		$A_1=$	$A_2=$

4) Plot absorbance Vs concentration for

    i) MnO_4^-/Co^{2+} solution at λ_1

    ii) MnO_4^-/Co^{2+} solution at λ_2

    iii) $Cr_2O_7^2/Ni^{2+}$ solution at λ_1

    iv) $Cr_2O_7^2/Ni^{2+}$ solution at λ_2

5) Calculate the unknown composition of the given mixture.

Calculation

[A]   Calculation of ϵ [for for $K_2Cr_2O_7$ and $KMnO_4$ system]

1. $(\epsilon_1)_{440} = (A_1)_{440}/C_1$
2. $(\epsilon_1)_{545} = (A_1)_{545}/C_1$
3. $(\epsilon_2)_{440} = (A_2)_{440}/C_2$
4. $(\epsilon_2)_{545} = (A_2)_{545}/C_2$

[B]   Calculation of concentration C_1 and C_2 in unknown mixture:

$$C_1 = \frac{(\epsilon_2)_{440}(A)_{440} - (\epsilon_2)_{440}(A)_{545}}{(\epsilon_1)_{440}(\epsilon_2)_{545} - (\epsilon_2)_{440}(\epsilon_1)_{545}}$$

$$C_2 = \frac{(\epsilon_1)_{440}(A)_{545} - (\epsilon_1)_{545}(A)_{440}}{(\epsilon_1)_{440}(\epsilon_2)_{545} - (\epsilon_2)_{440}(\epsilon_1)_{545}}$$

Where

$(A)_{440} = (A_1)_{440} + (A_2)_{440}$

$(A)_{545} = (A_1)_{545} + (A_2)_{545}$

[similarly we can calculate for $NiSO_4$ and $CoSo_4$ system]

Result:-

1)Concentration of $Cr_2O_7^{2}/Ni^{2+}$ in solution =_____

2)Concentration of MnO_4^-/Co^{2+} in solution =_____

discuss the various transition involved in terms of MO theory.

Polarimetry

Aim:

Determine the specific rotation of camphor in benzene and carbon tetrachloride by polarimetry.

Chemicals: Camphor, Benzene, Carbon tetrachloride

Procedure:

1) Prepare 10%, 5% and 2.5% solution of camphor in benzene and carbon tetrachloride separately
2) Fill the polarimeter tube with pure benzene(solvent) and measure angle of rotation.
3) Fill the polarimeter tube with 2.5% solution of camphor in benzene and ameasure angle of rotation. Similarly determine angle of rotation for 5% and 10% solution of camphor in benzene.
4) Repeat (2) by taking carbon tetrachloride as solvent.
5) Repeat (3) for 2.5%, 5% and 10% solution of camphor in carbon tetrachloride.
6) Calculate specific rotation of camphor in benzene and carbon tetrachloride using appropriate relation.
7) Specific rotation is calculated by using formula

$$[\,\alpha\,]_D^t = \frac{100 \times \alpha}{C \times l}$$

Where l = length of solution

C = Conc. In gm/ 100 ml of solution

Present your observations as follows.

Length of the polarimeter tube = ----------------- dm

Concentration of camphor solution (%) C	Solvent	Angle of rotation	Corrected angle of rotation	Specific rotation
2.5 5 10	Benzene			

2.5 5 10	Carbon tetra chloride			

8) Comment on the effect of solvent on specific rotation of camphor.

Aim:- To Determine the percentage of two optically active substances(d-glucose &

d tartaric acid) in a mixture polarimetrically

Theory

When the two substances do not interact, the specific rotation of two optically active substance in the solution are additive. If C_1 and C_2 are the weight fraction of the two solutes in the solution and $[\alpha]_1$ and $[\alpha]_2$ are their respective specific rotations,then

$[\alpha]_{12} = C_1 [\alpha]_1 + C_2 [\alpha]_2 = C_1 [\alpha]_1+(1- C_1) [\alpha]_2$

Where $[\alpha]_{12}$ is the specific rotation of the mixture.

From above relationship, we get

$$C_1 = \frac{[\alpha]_{12} - [\alpha]_2}{[\alpha]_1 - [\alpha]_2}$$

And $C_2 = (1- C_1)$

Chemicals :-

d-glucose & d tartaric acid ,Distilled water

Procedure:-

1) Prepare 5% and 2.5% solution of d-glucose and d-tartaric acid separately in distilled water. Filter the solution.
2) Determine the angle of rotation of each solution and hence specific rotation.
3) Prepare 5% solution of given mixture and determine its angle of rotation and hence specific rotation.
4) Specific rotation is calculated by using formula

$$[\alpha]_D^t = \frac{100 \times \alpha}{C \times l}$$

Where l = length of solution

C = Conc. In gm/ 100 ml of solution

5) Percentage of glucose = C_1
& % of d- tartaric acid = C_2 are determine by using formula

$$C_1 = \frac{[\alpha]_{12} - [\alpha]_2}{[\alpha]_1 - [\alpha]_2}$$

Observation & Calculation:-

Sr. No.	Substance	Angle of rotation	Specific rotation α
1	d-glucose		α_1
2	d-tartaric acid		α_2
3	Mixture		α_{12}

Result:-

Percentage of optically active substances =_____%

NON INSTRUMENTAL

Chemical Kinetics

Aim :

To Investigate kinetics of iodination of acetone

Chemicals :-

0.1 M Iodine, 1 M aqueous Acetone, 1 M H_2SO_4, 0.5 N $NaHCO_3$, 0.01 M $Na_2S_2O_3$, Starch indicator

Procedure:-

Set I:

1) Take the following solutions in two dry bottles.

   i)   20 ml of 0.1 M Iodine

   ii)   10 ml of Acetone + 20 ml of 1 M H_2SO_4 + 50 ml of water

2) Keep both the bottles in water bath for 10 minutes. Mix the two solutions at once and note the time of mixing.

3) Pipette out 10ml of reaction mixture in a titration flask containing 10 ml of 0.5 N $NaHCO_3$

4) Titrate the mixture against 0.01 M $Na_2S_2O_3$ at the time intervals of 5, 10, 15, 20, 25 and 30 minutes from the start using starch as an indicator.

Set-II

   i)   25 ml 0.1 M Iodine

   ii)   10 ml of 1 M Acetone + 20 ml 1 M H_2SO_4 + 45 ml water

5) Repeat (2) to (4) for this set also

6) Present your observation as follows

   Set:..........

Time `t` minute	Titre reading
5	
10	
15	
20	
25	
30	

7) Plot titre reading Vs t for both sets. Find the slopes.

8) Comment on the order of reaction from the slope of the graph

Graph: Plot a graph of titrant volume against time, for two sets. These plots will be straight lines slopping down with constant value of slopes (K)

Result:-

Comment on the order of reaction from the slope of the graph

Aim :

To determine Energy of Activation of Hydrolysis of Methyl Acetate in presence of HCl (calculation and graph are expected from excel programming).

Requirements

Methyl acetate, 0.5N HCl, 0.1N NaOH, phenolthalein indicator, ice water.

Procedure

Set A at Room temperature = T_1^0 C

1. Fill the burette with 0.1N NaOH
2. Take 50ml of 0.5N HCl in stopper bottle and 5ml methyl acetate in another bottle, place in water bath for 10minutes
3. Mix both the reagents, start the stop watch, record the time of mixing as zero minute
4. Pipette out 5ml of reaction mixture immediately, pour it in conical flask containing, 20ml ice water and two drops of phenolthalein indicator.
5. Titrate the reaction mixture with 0.1N NaOH
6. Record BR as V_0 at Zero time
7. Record BR at 10, 20, 30 and 40 minutes as V_{10}, V_{20}, V_{30} and V_{40} respectively.
8. Ask for V_∞
9. Calculate Rate constant (k_1) by Excel Programming.

Set B at temperature T_2^0C= $(T_1+5)^0$C

1. Repeat all the above steps at higher temperature.
2. Calculate Rate constant k_2 at higher temperature by excel programming

Calculations by Excel programming:

Double click below excel file for sample calculations

Calculations from
excel.xlsx

Set A at room temperature T_1^0C

V_∞=.....ml   a=(V_∞- V_0)ml

1. Open excel sheet, write title of observation table in Row 1
2. Put the values of Time(min) in column A and BR(ml) in column B
3. Select cell C3, give formula, =(B3 – V_0), drag it to the last, which gives values of x at different time intervals
4. Select cell D3, give formula, =(a – C3), drag it to the last, which gives values of (a-x)
5. Select the cell E3, give the formula, =log(a/D3), drag it to the last which gives values of log(a/a-x)
6. Select the cell F3, give the formula, =(2.303xE3)/A3, drag it to the last which gives values of rate constant at different interval of time

7. Select all values rate constant in column F, obtain mean
$k_1 = (\sum k_{10} + \ldots \ldots + k_{40})/4$

Set B at temperature, $T_2 = (T_1 + 5)^0C$

Repeat all steps of Set A by using excel sheet,

The mean rate constant will be k_2

Graphs

Set A

1. Select the values of column A(ie Time) and values of columnE3 (ie log(a/a-x)
2. Insert XY scatter, select suitable line pattern, graph will be obtained.
3. Delete horizontal lines by right click on one of the lines
4. Put the cursor on line(appr. straight line), right click, select Add trendline and equation
5. From the slope of the equation, calculate rate constant k_1

Set B

Repeat all the above steps for set B, obtain the values of rate constant k_2

Energy Activation

1. **By calculation**

$$Ea = \frac{2.303RT_1T_2}{(T_2 - T_1)} \log\left(\frac{k_2}{k_1}\right)$$

2. **From graph**

From the above equation and by using values of rate constants from graph calculate energy of activation.

Results
1. **Ea by calculation=**
2. **Ea by graph=**
3. **Attach print out of excel sheet**

NON INSTRUMENTAL

Aim :

To determine the solubility curve for water-acetic acid-chloroform system.

Chemicals: Glacial acetic acid, Chloroform, Distilled water

Procedure

1) Fill three clean burettes separately with chloroform, glacial acetic acid and distilled water.
2) Prepare 10%, 20%, 30% ……….90% chloroform in acetic acid (V/V) by taking chloroform-acetic acid mixture as (2ml+18ml), (4ml+16ml), (6ml+14ml), ……….(18ml+2ml) in different stoppered bottles/ titration flasks.
3) Titrate the mixture in bottle No 1 with distilled water till turbidity just appears. Note the reading.
4) Repeat (3) for other bottles and record the volume of water.
5) Calculate the percentage composition of each component in the mixture of each bottle.

   Present your observations as follows.

Bottle No	ml of $CHCl_3$	ml of acetic acid	ml of CH_3COOH	% $CHCl_3$	% CH_3COOH	% H_2O
1	2	18				
2	4	16				
3	6	14				
.	.	.				
.	.	.				
.	.	.				
9	18	2				

Calculations:

Densities

Water=1.0 g/cc, Acetic acid=1.05 g/cc, Chloroform=1.478 g/cc

Calculate % acetic acid, Chloroform and Water as follows:

Wt of acetic acid = ml of acetic acid x Density of acetic acid

Wt of Chloroform = ml of Chloroform x Density of Chloroform

Wt of Water= ml of Water x Density of Water.

Total weight = Wt of acetic acid + Wt of Chloroform + Wt of Water

$$\% \text{ Acetic acid} = \frac{\text{Wt of acetic acid}}{\text{Total weight}} \times 100$$

$$\% \text{ Chloroform} = \frac{\text{Wt of Chloroform}}{\text{Total weight}} \times 100$$

$$\% \text{ Water} = \frac{\text{Wt of Water}}{\text{Total weight}} \times 100$$

Graph:

Plot the percentage composition of each component on a triangular diagram and draw the solubility curve of the three component system.

Aim:-

Determination of partial molar volume of a component of a (mixture) ethanol in dilute aqueous solution.

Chemicals:- pyknometer / density bottle , thermostat, absolute ethanol

Procedure:-

1. Prepare a series of 25 ml solution of ethanol and distilled water in the ratio 0, 20, 40, 60, 80, 100 % by weight of accurately measuring the two components in the glass stopper bottle as shown below.

Sr. NO.	Weight % of ethanol	Volume of	
		Ethanol in ml	Water in ml
1	0	0.0	25
2	20	6.0	19
3	40	11.5	13.5
4	60	16.3	8.7
5	80	20.9	4.1
6	100	25	0.0

2. Determine the density of pure ethanol, distilled water and density of solution at 25^0C.

Observations:-

Sr. No.	Percentage of		Density	Sp. Vol. (Vsp)	Partial molar volume	Mole fraction of ethanol
	Ethanol	Water				
1	00	100				

2	20	80				
3	40	60				
4	60	40				
5	80	20				
6	100	00				

Calculation and Graphs

Calculate specific volume (reciprocal of density) of pure ethanol, water and each of the   solution.

Calculate number of moles and mole fraction of ethanol at each composition.

$$\text{No. of moles of ethanol} = \frac{\text{Weight of ethanol}}{\text{Molecular weight of ethanol}}$$

$$\text{No. of moles of water} = \frac{\text{Weight of water}}{\text{Molecular weight of water}}$$

$$\text{Mole fraction of ethanol} = \frac{\text{No. of moles of ethanol}}{\text{Total No. of moles}}$$

Plot a graph of % weight of ethanol on X axis against Specific volume on Y axis.

Draw a smooth curve through the point & draw tangents to curve at different concentration

The intercept of these tangents on 0% & 100% on Y axis gives the partial sp. Volume of water and ethanol respectively at these concentrations.

Calculate partial molar volume at various concentrations of water and ethanol by multiplying their partial sp volume by respective molecular weight.

Finally plot the partial molar volume against composition in mole fraction of ethanol on X axis and obtain the curve showing variation of partial molar volume with the mole fraction.

Result:-

Partial molar volume of a component of a (mixture) ethanol in dilute aqueous solution. = _____

Aim:

Study the effect of addition of an electrolyte on solubility of an organic acid

Chemicals: Solid organic acid (Benzoic acid /Salicylic acid), 0.02 N NaOH, Solid electrolyte (KCl, NaCl, NH_4Cl, Na_2SO_4, K_2SO_4 etc).

Procedure:

1. Prepare about 400 ml saturated solution of Benzoic acid /Salicylic acid at a temperature about 10^0 C above room temperature. Allow the solution to cool down to room temperature with constant stirring and filter the solution.
2. Pipette out 50 ml of saturated solution in five dry conical flasks.
3. Add 1,2,3,4 and 5 gm of given electrolyte in different flasks. Shake and allow to stand for 15 minutes.
4. Fill the burette with 0.02 N NaOH.
5. Determine the solubility of benzoic acid /Salicylic acid without addition of salt (Flask A) by titrating 10 ml of saturated solution with 0.02 N NaOH using phenolphthalein as an indicator.
6. Filter each mixture separately in beaker. Titrate 10 ml of these solutions against 0.02 N NaOH using phenolphthalein as an indicator. Determine the solubility of the acid in each solution.
   Present your observations as follows

Flask No	Amount of Electrolyte (gm)	Volume of Filtrate (ml)	Volume of NaOH (B.R.)				Solubility of acid (gm/100 gm of solvent)
			i	ii	iii	Const B.R.	
A	0	10					
B	1	10					
C	2	10					
D	3	10					

E	4	10					
F	5	10					

Calculations:

$$\text{Solubility of acid} = \frac{\text{Normality of NaOH x Volume of NaOH x Eq. Wt of NaOH}}{\text{Volume of acid solution}} \quad \text{g/l}$$

Hence calculate the solubility of the acid per 100 gm of solvent.

Graph:

Plot a graph of solubility of acid in gm/100 gm of solvent against the concentration of salt added.

Comment on the effect of addition of an electrolyte on solubility of an organic acid from the nature of graph.