RPS DEGREE COLLEGE
BALANA (MAHENDERGARH)-123029

Lab Manual

(Chemistry5th & 6th Semester)

Department of Chemistry

Syllabus

Distribution Law

(i) To study the distribution of Iodine between H2O and CCl4
(ii) To study distribution of benzoic acid in benzene and water. To study the equilibrium constant of complex reach e.g. I- I2 –I3

Conductometric Titration

(i) Determine of cell constant of the conductivity cell

(ii) (ii) Determination of solubility and solubility product of the given sparingly soluble salt.

Potentiometric Titration:

(i) Potentiometric titration of strong/weak and against weak/strong base.

Specific Rotation (Polarimetery)

(i) To determine the specific rotation of the given optically active compound.

Colorimetry:

(i) To verify the Lembert Beer's Law using KMnO4 K2Cr2 O7 solution.

EXPERIMENT 1

Aim: To determine distribution coefficient of I_2 between CCl_4 and water.

Apparatus: Reagent bottle, Burette, Pipettes, Conical flasks & measuring jar

Chemicals: Saturated I_2/CCl_4, M/20 Hypo, M/600 Hypo Starch indicator

Principle: When a common solute is added to a system of two immiscible liquids, solute distributes itself in a definite concentration ratio such way that its concentration ratio is constant at constant temperature irrespective of the amount of solute is added.

The Distribution coefficient is given by

$$K_D = \frac{Caq}{Corg}$$

$$\log Ca_q = \log K_D + \log C_{org}$$

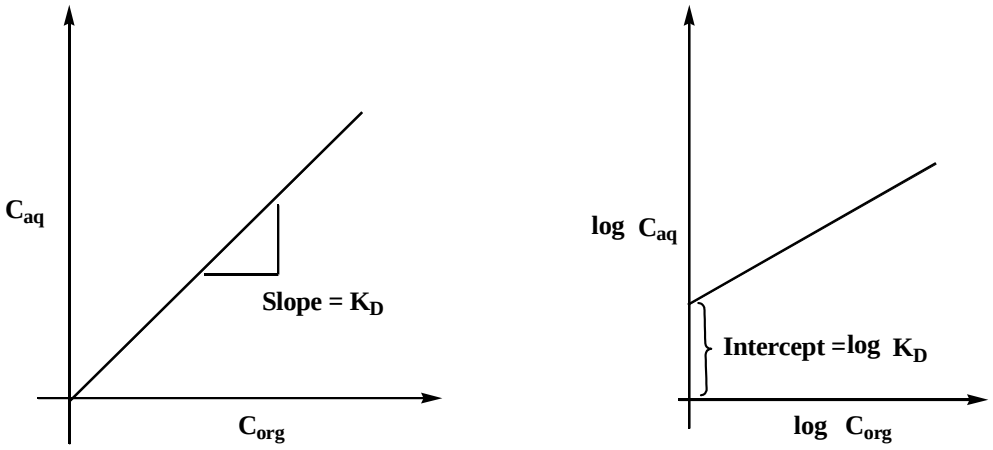
Where C_{org}&$C_{aq}$are concentrations of solute in organic & aqueous layers respectively

Procedure: Take 25mL of I_2/CCl_4 and add 25mL of distilled water in a clean reagent bottle. Shake vigorously about 15 minutes for proper distribution. Allow the reagent bottle for the layers to separate. Meanwhile fill the burettes with M/20 Hypo M/600 Hypo. Now pipette out 10mL of organic (lower) & 10mL of aqueous (upper) layers into two conical flasks separately. Titrate organic layer with M/20 Hypo solution and titrate aqueous layer with M/600 Hypo solution by adding 2-3 drops of Starch indicator. Note down the readings as V_{org}&V_{aq}respectively.

Now add 10mL of CCl_4& 10mL of distilled water to the reagent bottle. Shake vigorously about 10-15 minutes for proper distribution of solute between two layers. Do the titrations as described above. Repeat the same experimental procedure for both the layers (4-6 times) and then calculate concentrations of Iodine as C_{org}&C_{aq}respectively.

Note: Density of Water = 1.028 gm/ml; Density of CCl_4 = 1.59 gm/ml

Model graphs: Plot a graph between C_{aq} and $\mathbf{C_{org}}$, straight line passing through origin is obtained. From the slope distribution constant (K_D) can be calculated.



Model tabular form:

S.No	V_{org}	V_{aq}	C_{org}	C_{aq}	log C_{aq}	log C_{org}	KD = Caq /Corg

1							
2							
3							
4							
5							

Result:.

Nernst Distribution law is verified.

K_D (From calculations) = ……

K_D value from Graph -1 =……

K_D value from Graph - 2 = …….

EXPERIMENT 2

DETERMINATION OF CELL CONSTANT

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

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

Chemicals: N/10 KCl Solution

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

Specific conductance / measured conductance = Cell constant

Or Specific conductance = measured conductance x Cell constant.

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

Taking reciprocals 1/R = 1/p x a/1

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

Therefore specific conductance = conductance x 1/a

Therefore cell constant = 1/a

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

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

S. No.	Concentration	Observed conductance	Specific conductance	Cell constant

Result: The cell constant was observed to be --------

Experiment – 3

DETERMINATION OF SOLUBILITY PRODUCT OF SPARINGLY SOLUBLE SALT

Aim: To determine the solubility product of sparingly soluble salt using conductometer.

Apparatus:100ml beaker, conductometer

Chemicals required: 0.1M AgNO3, 0.1M KCl.

Principle:The salts which are soluble only to a very little extent are knowquantity that is dissolved in a saturated solution may be regarded as present at infinite dilution as sparingly soluble salts. Ex: AgCl, BaSO4, PbSO4 etc... As the solubility of sparingly soluble salt is very low, the small n. Thus, its equivalent conductance may be taken as conductance at infinite dilution, $\lambda\infty$.

Suppose the solubility of salt is 'S' gram equivalents per liter, then the volume which will contain 1 gram equivalents of the salt will be 1000mL. Now the equivalent conductance at infinite dilution is given by:

$\lambda_o = \lambda_v = \frac{1000Kv}{s}$

$$s = \frac{1000Kv}{\lambda v}$$

The specific conductance (kv) of the solution is known by determining the conductance and subtracting it from that of water.

True or observed conductance CT = $C_{solution}$-C_{water}

Here, kv = CT * Cell constant.

By determining the solubility of a sparingly soluble salt, one can determine the solubility product of AgCl by substituting it in the equation

$$Ksp = S*S = S2$$

Procedure: In100ml beaker, take about 25ml of 0.1M AgNO3 and 25ml of 0.1M KCl. Stir well. Note down the observed conductance.

Model tabular form:

S.No	Conductance (ms)	Specific conductance (k_v)	Solubility (S)	K_{sp} = Solubility product $(mol/lit)^2$

RESULT:- The solubility product of AgCl was determined to be:

Experiment – 4

Titration of a strong acid with a strong base – Titration of HCl against NaOH

Aim: To find out the concentration of the given hydrochloric acid solution by potentiometric method.

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

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

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

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

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

Procedure: Pipette out 10 ml of the given HCl solution into a clean beaker and dip the calomel electrode and the Platinum electrode. Now a pinch of quinhydrone is added to the HCl solution. Connect the two electrodes to a potentiometer to read the cell EMF. Fill the burette with the given NaOH solution (0. 1M). Before the addition of sodium hydroxide to the acid solution measure the EMF. Now add 1 ml of the given sodium hydroxide solution to the HCl solution present in the beaker and stir with a glass rod and measure the EMF. Continue the addition of equal volumes of base until a large change in EMF is observed. Now add 0. 2 ml of NaOH every time to get an accurate end point. Continue this addition until you get a minimum of 7-8 readings after the end point.

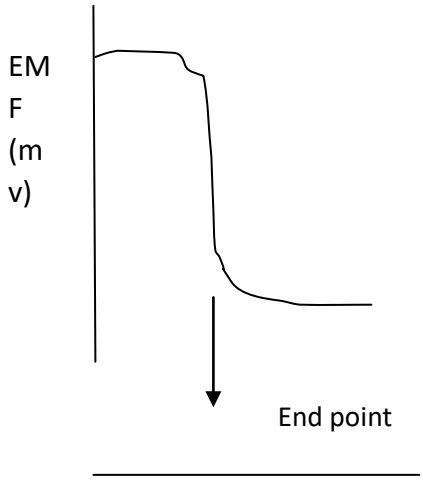
Plot a graph between the measured EMF of the cell and volume of sodium hydroxide where in a sigmoid curve will be obtained. The inflexion of the curve

i.e, where the curve changes its direction is taken as the end point of the titration. Accurate endpoint can be obtained by plotting a graph between □E/□V against Vavg.

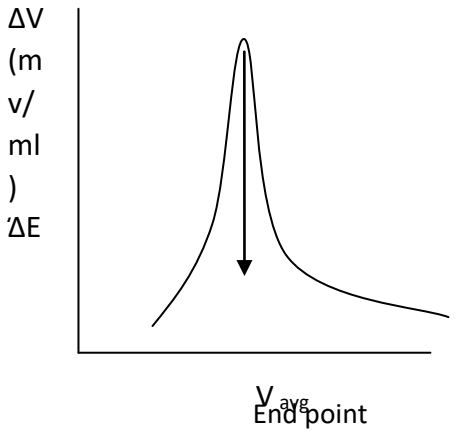
Result: Concentration of the given HCl solution = _____M

S. NO	Volume of NaOH added (ml)	EMF (mv)	ΔE	ΔV	ΔE / ΔV	Vavg	$\frac{pH = 0.4595 - E_{cell}}{0.0591}$

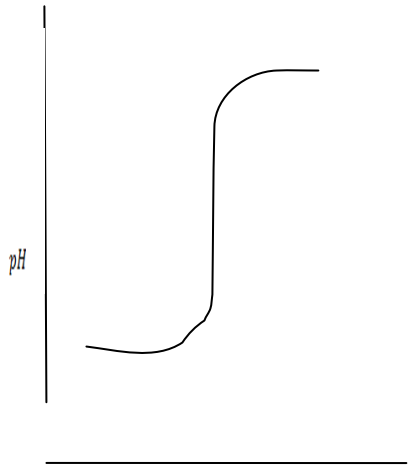
EMF Vs Volume of NaOH



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



pH Vs Volume of NaOH



CALCULATIONS:

HCl Vs NaOH

$$M1V1 = M2V2$$

M_1 = Molarity of the sodium Hydroxide solution
= 0.1M

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

M_2 = Molarity of the given HCl solution

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

$$M_2 = M_1V_1 / V_2$$

$$M_2 = 0.1 \times E.P.V / 10$$

Experiment – 5

Determination of Specific rotation of Sucrose

Aim: To determine the specific rotation of cane sugar (sucrose).

Apparatus: Polarimeter, sodium lamp, Polarimeter tube, standard flasks, simple balance, pipette, etc.

Chemicals: 20% Sucrose solution.

Principle: Specific rotation can be defined as the angle of rotation when polarized light is passed through one decimeter of the solution having concentration of 1g/1ml. It is represented by α.

$$[\alpha] = \frac{\theta}{lc}$$

Where θ = *angle of rotation*

l = length in decimeters

c = Concentration of the solution

When 'c' is expressed in %, then

$$[\alpha] = \frac{\theta \times 100}{lc}$$

When l = 2 decimeters then, $[\alpha] = \frac{\theta \times 50}{c}$

On rearranging, $\theta = c \times \frac{\alpha}{50}$

Thus, when a graph is plotted between angle of rotation and concentration a straight line passing from the origin is obtained. From the slope of the straight line specific rotation can be evaluated.

Procedure: Calibration of the polarimeter tube

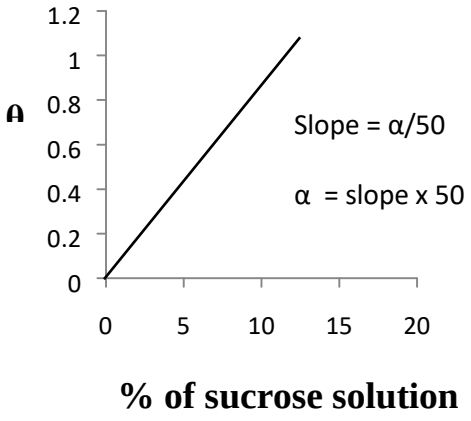
Set up a sodium vapour lamp and adjust the height of the lamp and the optical axis of the instrument for maximum illumination of the polarizer. Take a properly cleaned polarimeter tube and fill it with distilled water.

Avoid entry of air bubbles into the tube. There should be no liquid drops on the outside of the glass plate. Place the tube between the polarizer and analyzer and determine the zero point of the polarimeter by rotating the analyser until two halves of the field of view are equally dark.

By weighing 20 grams of sucrose and dissolving in 100ml of water in a clean standard flask results in 20% sucrose solution. The obtained solution is filled in the polarimeter tube and the angle of rotation of 20% sucrose solution is measured. The experiment is repeated with 15%, 10%, 5% and 2.5% concentrations of sucrose solution.

A graph is plotted between θ and % of sucrose solution. A straight line is obtained. From the slope specific rotation can be obtained.

Result: The specific rotation of sucrose is ……..deg.$dm^{-1}g^{-1}cm^{3}$



S. No.	% of sucrose solution	θ (Angle of rotation)

Experiment – 6

VERIFICATION OF BEER'S LAW USING $CuSO_4$ SOLUTION

Aim: To determine the concentration of given $CuSO_4$ solution by verifying the Beer's law using colorimeter.

Apparatus: Colorimeter, graduated pipette, standard flask, test tubes, cuvettes etc.

Chemicals required: 0.2M $CuSO_4$ solution, distilled water

Principle: Beer's law is applicable to only to dilute solutions. It states that when a monochromatic light is passed through a solution intensity of light decreases with thickness and this decrease in intensity is directly proportional to the intensity of incident light and the concentration of the solution.

$$I = I0\ e^{-\varepsilon\ c\ x}$$

Where I = Intensity of the transmitted light; I_0 = Intensity of the incident light C = Concentration of the solution; x = Path length of the cuvette ε = Molar extinction coefficient

$$I / I_0 = e^{-\varepsilon cx}, \qquad \ln(I/ I_0)= -\varepsilon cx$$

The value of ε (epsilon) depends on the nature of the absorbing solute but independent of the concentration of the solution. Decrease in the intensity of the transmitted light is due to the absorption of light, which may lead to photochemical reactivity.

In colorimetric studies transmittance is defined as the fraction of light passes through the sample

$$\% \text{ Transmittance (T)} = (I / I_0) \times 100$$

A more useful and convenient quantity in performing analysis is the absorbance or negative log of transmittance.

Absorbance or Optical density= A= log (1/T) = - log (T)

A linear relation exists between absorbance (O. D) and concentration of the sample is known as Beer's law.

Absorbance or Optical density= A= log (I_0/I) = εcx

Before verification of Beer's law, it is necessary to a select a suitable wave length and determines whether Beer's law is valid at the wavelength selected. The most suitable wavelength is that at which maximum absorbance is observed, called λ_{max}. The λ_{max}will always be at the same wavelength for a given solution under any condition.

Procedure: Colorimeter is switched on, wait for 10-15min so that the instrument acquires temperature stability. The intensity of the light from the lamp depends on the environmental temperature. Now transmittance is adjusted to 100% or O.D is adjusted to zero with distilled water (Since water is the solvent used in the preparation of $CuSO_4$ solution), which is taken in a cuvette. By using various filters the λ_{max}value of the $CuSO_4$ solution (Bluish green colour solution) can be determined. Different concentrations of $CuSO_4$ solutions are prepared by mixing 10,9,8,7,6,5,4,3,2,1 ml $CuSO_4$ solutions from the stock with 0,1,2,3,4,5,6,7,8,9 ml of distilled water respectively. The O.D value is measured for each set at the λ_{max} which is in the range of 650-700nm (Red colour filter).

Model tabular form: a) Filter selection

S.No	Wavelength(nm)	Optical density
1		
2		
3		
4		

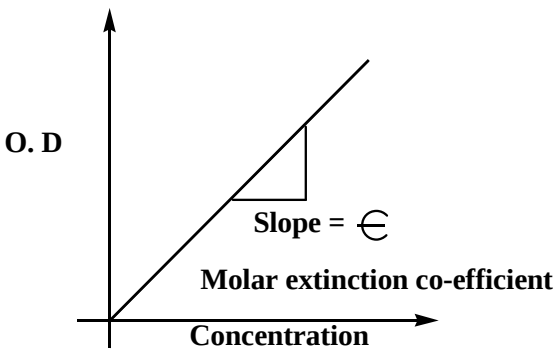
5		

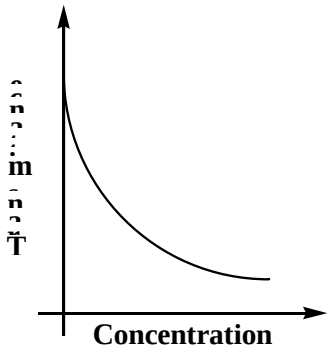
b) <u>Beer's law verification</u>

S. No	**Volume of $CuSO_4$ (ml)**	**Volume of water(ml)**	**Concentration(mol/lit)**	**Optical density**
1.	**9**	**1**	**0.18**	
2.	**8**	**2**	**0.16**	
3.	**7**	**3**	**0.14**	
4	**6**	**4**	**0.12**	
5	**5**	**5**	**0.10**	
6	**4**	**6**	**0.08**	
7	**3**	**7**	**0.06**	
8	**2**	**8**	**0.04**	
9	**1**	**9**	**0.02**	

<u>Model graph</u>: A graph is plotted between optical density and concentration of $CuSO_4$ .A straight line passing through the origin is obtained. Slope of the line is equal to Molar extinction co-efficient.

Calibration curve





Result: A straight line passing through origin is obtained in the graph. Hence, Beer's law is verified.

The molar extinction co-efficient = …….. Lit mol^{-1} cm^{-1}

Experiment – 1

Aim: To determine the strengths of strong (HCl) and weak acid (AcOH) in a given mixture conductometrically.

Apparatus: Conductivity Bridge, Conductivity cell, beaker, Pipette, Micro burette, Glass rod

Chemicals required: 0.1M HCl, 0.1M CH_3COOH, 0.5M NaOH

Principle: Conductometric titration is a type of titrationin which the electrolytic/ionic conductivityof the solution continuously monitored as one reactantis added. The principle of conductometric titration is based on the fact that during the titration, one of the ions is replaced by the other and invariably these two ions differ in the ionic conductivity with the result that conductivity of the solution varies during the course of titration. The main advantages to the conductometric titration are its applicability to very dilute, homogeneous suspension, coloured solutions and to system that involve relative incomplete reactions, which cannot be used with normal indicators.

The conductance method can be employed to follow the course of a titration, provided that there is a significant difference in conductance between the original solution and the reagent of the product of reaction. It is not necessary to know the cell constants, since relative values are sufficient to permit locating the equivalence point. The conductance produced by an ion is proportional to its concentration (at constant temperature), but the conductance of a particular solution will in general not vary linearly with added reagent, because of the dilution effect of water being added along with reagent added.

The conductivity of the solution is inversely proportional to the size of the ions .if the size of the ions is increasing then the conductivity of the solution will decrease because the mobility of the ions will decrease by increasing the size of the ions. By increasing the temperature, the mobility of the ions in the solution will increase. So temperature has a direct effect on conductance of solution.

Upon adding a strong base to a mixture of strong acid and weak acids the following reactions occur sequentially

$$[H^+ + Cl^-] \quad + [Na^+ + OH^-]\ Na^+ + Cl^- + H_2O$$

$$CH_3COOH + \quad [Na^+ + OH^-]\ CH_3COO^- + Na^+ + H_2O$$

According to Kohlrausch law, the electrical conductance of a solution depends upon number/ concentration, mobility/speed of ions and temperature. As the titration proceeds, first strong acid reacts or neutralises with base and the conductance of the solution gradually decreases. This continues until the neutralization of strong acid. Further, weak acid reacts with the strong base, conductance increases slowly. After fully neutralization weak acid causes sudden increase in conductance. From the sharp break in the curves, equivalence points can be determined, from which the strength of the acids can be calculated

Strong base (NaOH) neutralizes the strong acid (HCl) first rather than weak acid (AcOH) indicated by decrease in conductance. This is due to comman ion effect. Due to this, dissociation of weak electrolyte is suppressed. Hence, AcOH remains un-neutralized till the complete neutralization of strong electrolyte (HCl). Then start the neutralization of AcOH indicated by increase in conductance values. When the acid mixture is completely neutralized, further addition of base will results the increase in conductance.

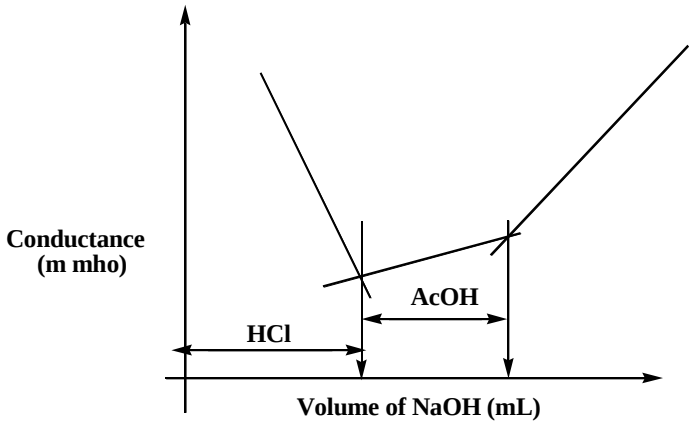
Procedure: Take 25 mL of 0.1M HCl and 25 mL 0.1M AcOH in a clean 100 mL beaker. Connect the conductivity cell to the conductivity meter. Once the conductivity meter is standardized, add 1mL of 0.5M NaOH from the burette to solution (acid mixture) containing beaker. Stir the solution carefully and note down the corresponding conductance value. Continue the addition of NaOH solution from the burette and record the conductance after every addition and tabulate the data.

Tabular form:

S. No	Volume of NaOH (mL)	Conductance (m mho)
1	0	
2	1	
3	2	
4	3	
5	4	
6	5	

Model graph:

Plot a graph between conductance values against the volume of the NaOH added. Three straight lines are obtained. The intersection of the first two lines gives the end point of strong acid and the intersection of the second and third lines gives the end point of the weak acid.



Result:

The volume of NaOH (0.5M) required for the strong acid in a given mixture =……mL

The volume of NaOH (0.5M) required for the weak acid in a given mixture =……mL

Strength/Concentration of given HCl solution is …….M

Strength/Concentration of given AcOH solution is …….M

Experiment – 2

AIM:-To determine the CST of phenol – water system.

APPRATUS USED :- A boiling tube, a stirrer, a thermometer graduated to 0.1 degree, 400ml beaker, iron stand, tripod stand.

CHEMICALS REQUIRED:-Phenol and distilled water.

OBJECTIVE:-It is based on the fact that when water and phenol are mixed together at room temperature they form heterogeneous mixture having white turbidity. Now when the system is heated a temperature comes where turbidity disappears on cooling turbidity appears again. Noting the temperature at which turbidity disappears and at which it appears the mean of the two temperatures give the temperature of mixing of phenol water system. Now the solutions of phenol od different compositions are prepared and the temperature of miscibility of the two is determined in each case. Then a graph of composition of mixture vs. miscibility temperature is plotted. The temperature corresponding to the maximum is the CST of the system.

PROCEDURE:-

(I)Take 60gm of phenol in a previously weighed boiling tube.

(ii)Add 2ml of distilled water with the help of a graduated pipette into the phenol taken in a boiling tube. Thus the % of phenol by weight is 75%.

(iii)Fill 2/3rd of the 400ml beaker with water and keep it on the wire gauge placed on the tripod stand.

(iv)Clamp the boiling tube into the beaker as shown. Fit the cork with two holes, one for the stirrer and other for thermometer.

(v)Heat the beaker slowly and stir the phenol water mixture. Note the temperature at which turbidity just disappears. Stop heating. Now allow the mixture to cool and note the temperature when the turbidity just appears.

(vi)Now again add 2ml of distilled water with the help of a graduated pipette. Thus the % of phenol by weight is 60%. Repeat step (v).

Repeat the process after adding 2ml of distilled water each time taking at least seven or eight readings.

OBSERVATIONS:-

Weight of empty boiling tube = $w_1$gm

Weight of tube + phenol = (w_1 + 6)gm

Weight of phenol = 6.0gm

Density of water = 1gm/ml (assuming)

RESULT:-

(i)CTS of phenol-water system = ……..^{o}C

(ii)Composition of the system

Phenol = ……..%

Water = ……%

PRECAUTIONS:-

(i)Handle phenol very carefully as it causes severe skin burns.

(ii)Take care that in each case the level of phenol-water system in the tube must be atleast one cm below the level of water in the beaker.

(iii)For gradual and more uniform heating, surrounded the boiling tube with outer jacket (a more bigger tube).

(iv)Stirring inside the solution and outside in water must be done constantly.

(v)The bulb of the thermometer must remain dipping in phenol water system.

Experiment – 3

Aim: To find out the strength of the given ferrous ammonium sulphate solution by titrating it against potassium dichromate solution potentiometrically.

Apparatus: Potentiometer, standard cell, SCE, Pt wire, 100mL beakers, pipette

Chemicals Required: 0.1N Ferrous ammonium sulphate (FAS), 0.1N Potassium dichromate (M/60 $K_2Cr_2O_7$), 6N H_2SO_4, H_3PO_4

Principle: Potentiometric methods of analysis are based upon measurements of the potential of electrochemical cells under conditions of zero current, where the Nernst equation governs the operation of potentiometry.

Redox reaction involved is given below

$$\mathbf{6Fe^{2+} \rightarrow 6Fe^{3+} + 6e^{-}(Oxidation)E^{O} = 0.77V}$$

$$\mathbf{Cr_2O_7^{-2} + 14H^{+} + 6e^{-} \rightarrow 2Cr^{3+} + 7H_2O(Reduction)E^{O} = 1.33V}$$

Overall reaction: $\mathbf{6Fe^{2+} + Cr_2O_7^{-2} + 14H^{+} \rightarrow 6Fe^{3+} + 2Cr^{3+} + 7H_2O}$

Above reaction involves $6e^-$ transfer between Fe^{+2} and Cr^{+6}

The cell can be depicted as follows

Hg (l), $Hg_2Cl_{2(S)}$ |KCl || Fe^{+2}, Fe^{+3}, Pt Pt (or) SCE || Cr^{+6}, Cr^{+3}, Fe^{+2}, Fe^{+3},

$$\mathbf{E_{cell} = E_{Fe} - E_{SCE}}$$

$$E_{Fe} = E^{0}_{Fe} + 0.0591 \log \frac{[Fe^{3+}]}{[Fe^{2+}]}$$

$$E_{Fe} = E^{0}_{Fe} + 0.0591 \log \frac{[Fe^{3+}]}{[Fe^{2+}]} - 0.242$$

As the titration proceeds, i.e. when the $K_2Cr_2O_7$ is added to ferrous ammonium sulphate, ferric (Fe^{+3}) concentration increases and ferrous (Fe^{+2}) concentration decreases. Therefore, half-cell potential and the cell potential increases. Near the end point the rate of change in potential will be maximum as the $[Fe^{+3}]/[Fe^{+2}]$ ratio changes significantly. On crossing the equivalence point, EMF changes in small increments and finally it reaches saturation. This is due to, after equivalence point, only Fe^{+3} ions are present and since no Fe^{+2} ions are present in solution. After end point the cell potential is governed by other redox couple $[Cr^{+6}]/[Cr^{+3}]$

Actually the electrode potential depends on the concentration of H^+ ions besides the concentration of Fe^{+2} and Fe^{+3}. Therefore, to avoid the effect of change in the $[H^+]$ on the electrode potential, this titration is carried out in presence of large amount of $[H^+]$. Hence the cell potential is depends on concentrations of Fe^{+2}/Fe^{+3} ratio. Acidic solutions such as H_2SO_4 and/ or H_3PO_4 are used to avoid hydrolysis of Fe^{+2}. Phosphoric acid forms complexes with Fe^{+3}, lowering the concentration of simple Fe^{+3} ions and decreasing the potential of the Fe^{+3}/Fe^{+2} couple.

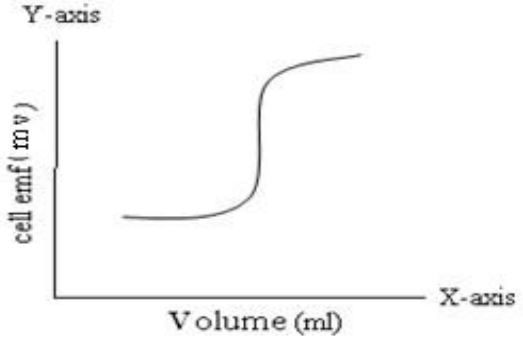
Procedure: Take 20mL of 0.1N ferrous ammonium sulphate (FAS) in a 100mL beaker, add 5mL of 6N H_2SO_4 or [H_3PO_4 + H_2SO_4 mixture] to the beaker and add sufficient amount of distilled water (25 mL) so that the electrodes are completely dipped in the solution. Combine the Pt electrode (contact electrode) with the calomel electrode through a salt bridge. The two electrodes are connected to the potentiometer. Once the potentiometer is standardized, add 1mL of 0.1M $K_2Cr_2O_7$ from the micro burette to FAS taken in a beaker. Stir the solution carefully and note down the corresponding EMF value. Continue the addition of $K_2Cr_2O_7$ solution from the burette and note the EMF after every addition and tabulate the data

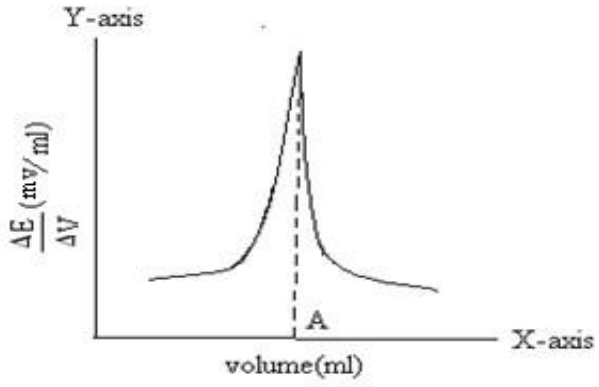
Model Tabular Form:

S. No	Volume of K2Cr2O7 (mL)	EMF (mv)	ΔE (mv)	ΔV (mL)	ΔE/ΔV
1	0				
2	1				
3	2				
4	3				
5	4				
0	5				

Graph: (1) Plot a graph between EMF (mV) and volume of $K_2Cr_2O_7$ (mL) added. A sigmoid type curve is obtained. From the graph equivalence point can be determined.

(2) Plot a graph between ΔE/ΔV and volume of $K_2Cr_2O_7$, a differential graph is obtained. From the graph equivalence point can be determined





Result: The end point for the titration of 20mL FAS against 0.1N $K_2Cr_2O_7$ is ……. mL

Strength/Concentration of given FAS solution is …….M

Experiment – 4

Aim: To determinethe concentration of given $KMnO_4$ solution by verifying the Beer's law using colorimeter.

Apparatus: Colorimeter, graduated pipette, standard flask, test tubes, cuvettesetc.

Chemicals required: $5x10^{-4}$M $KMnO_4$ solution, distilled water

Principle: Beer's law is applicable to only to dilute solutions. It states that when a monochromatic light is passed through a solution intensity of light decreases with thickness and this decrease in intensity is directly proportional to the intensity of incident light and the concentration of the solution.

$$\mathbf{I = I0\ e^{-\varepsilon\ c\ x}}$$

Where I = Intensity of the transmitted light; I_0 = Intensity of the incident light C = Concentration of the solution; x = Path length of the cuvette ε = Molar extinction coefficient

$$\mathbf{I / I_0 = e^{-\varepsilon cx}, \qquad ln(I/ I_0)= -\varepsilon cx}$$

The value of ε (epsilon) depends on the nature of the absorbing solute but independent of the concentration of the solution. Decrease in the intensity of the transmitted light is due to the absorption of light, which may lead to photochemical reactivity.

In colorimetric studies transmittance is defined as the fraction of light passes through the sample

% Transmittance (T) = (I / I_0) x 100

A more useful and convenient quantity in performing analysis is the absorbance or negative log of transmittance.

Absorbance or Optical density= A= log (1/T) = - log (T)

A linear relation exists between absorbance (O. D) and concentration of the sample is known as Beer's law.

Absorbance or Optical density= A= log (I_0/I) = εcx

Before verification of Beer's law, it is necessary to a select a suitable wave length and determines whether Beer's law is valid at the wavelength selected. The most suitable wavelength is that at which maximum absorbance is observed, called λ_{max}. The λ_{max}will always be at the same wavelength for a given solution under any condition.

Procedure: Colorimeter is switched on, wait for 10-15min so that the instrument acquires temperature stability. The intensity of the light from the lamp depends on the environmental temperature. Now transmittance is adjusted to 100% or O.D is adjusted to zero with distilled water (Since water is the solvent used in the preparation of $KMnO_4$ solution), which is taken in a cuvette. By using various filters the λ_{max}value of the $KMnO_4$ solution (Purple or violet colour solution) can be determined. Different concentrations of $KMnO_4$ solutions are prepared by mixing 10,9,8,7,6,5,4,3,2,1 ml $KMnO_4$ solutions from the stock with 0,1,2,3,4,5,6,7,8,9 ml of distilled water respectively. The O.D value is measured for each set at the λ_{max} which is in the range of 520-540nm (green colour filter).

Model tabular form: a) Filter selection

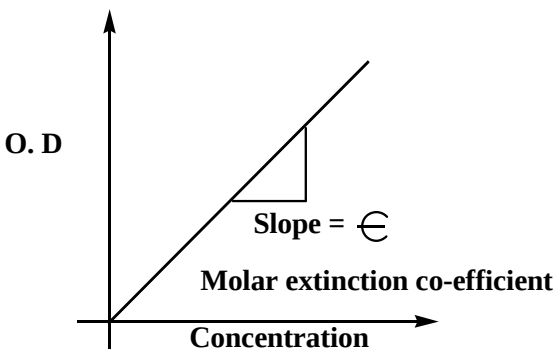
S.No	Wavelength(nm)	Optical density
1		
2		
3		
4		
5		

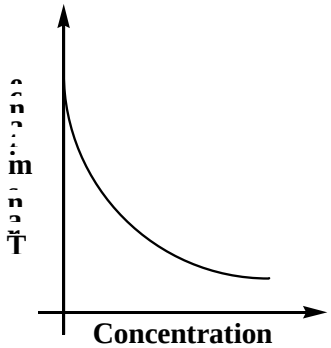
b) Beer's law verification

S.No	Volume of KMnO4 (ml)	Volume of water(ml)	Concentration(mol/lit)	Optical density
1.	9	1	4.5×10^{-4}M	
2.	8	2	4.0×10^{-4}M	
3.	7	3	3.5×10^{-4}M	
4	6	4	3.0×10^{-4}M	
5	5	5	2.5×10^{-4}M	
6	4	6	2.0×10^{-4}M	
7	3	7	1.5×10^{-4}M	
8	2	8	1.0×10^{-4}M	
9	1	9	0.5×10^{-4}M	

Model graph: A graph is plotted between optical density and concentration of $KMnO_4$. A straight line passing through the origin is obtained. Molar extinction coefficient can be determined from the slope of the line.

Calibration curve





Result: A straight line passing through origin is obtained in the graph. Hence, Beer's law is verified.

The molar extinction co-efficient = …….. Lit mol^{-1} cm^{-1}