KARPAGAM ACADEMY OF HIGHER EDUCATION
(Deemed University Established Under Section 3 of UGC Act 1956)
COIMBATORE-21

DEPARTMENT OF CHEMISTRY

Class	:	M.Sc Chemistry
Subject	:	Physical Chemistry Practical-I
Subject Code	:	17CHP311
Semester / Year	:	II/III

SYLLABUS

Semester-III

17CHP311 PHYSICAL CHEMISTRY PRACTICAL -I 4H 2C

(MOLECULAR WEIGHT DETERMINATION AND CONDUCTOMETRIC TITRATIONS)

Instruction Hours/week:L: 0 T:0 P:4 Marks: Internal:40 External: 60 Total:100

Scope
Physical chemistry practicals frame much of our understanding of the natural world and continue to bring new technologies that are useful to every aspect of human life. Physical chemistry practicals are an exciting and challenging course, which helps us to understand the various aspects of compounds used in our day to day life. This course presents the knowledge about thermal and conductometric titrations and solubility of a sparingly soluble salt.

Objectives
On successful completion of the course the students should have
1. Learnt about the thermal and conductometric titrations.
2. Learnt the principles of solubility of a sparingly soluble salt.

Methodology

Blackboard teaching and Demonstration.

Contents
Heat of solution from solubility.

Molecular weight determination by
- i. Freezing point depression of solvents benzene and water by Beckmann method
- ii. By Rast micro methods

Distribution of activity and activity co-efficient by freezing point method.

Distribution co-efficient and determination of equilibrium constant.

Conductivity experiments:

Determination of-

i) Equivalent conductance of a strong electrolyte and verification of Debye-Huckel Onsager law.

ii) Verification of Ostwald dilution law and Kohlraush law for weak electrolytes.

Conductometric determination of pK_a of a weak acid.

Hydrolysis constant of aniline hydrochloride.

Determination of the solubility of a sparingly soluble salt.

Conductometric titrations:

Acid-base and precipitation titrations including mixture of halides.

SUGGESTED READINGS:

Text Books:

1. Lepse, P. A., & Lyle B. P., (1986). *Lab Manual for Lingren's Essentials of Chemistry*. New Delhi: Prentice Hall.
2. Pandey, O. P, Bajpai, D. N., & Giri, S. (2001). *Practical Chemistry* (VIII Edition). New Delhi: S. Chand Publications.
3. Santi Rajan Palit and Sadhan Kumar, (1971). *Practical Physical Chemistry* (I Edition). Calcutta: Joy Publishers.

Reference Books:

1. Siddhiqui, Z. N. (2002). *Practical Industrial Chemistry* (I Edition). New Delhi: Anmol Publications Pvt. Ltd.
2. Thomas, A.O, (2003). *Practical Chemistry*. Cannanore: Scientific Book Center.
3. Venkateswaran, V., Veeraswamy, R., & Kulandaivelu, A. R. (2004). *Basic Principles of Practical Chemistry* (II Edition). New Delhi: S. Chand Publications.

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Coimbatore – 641 021.

LESSION PLAN
DEPARTMENT OF CHEMISTRY
M.Sc CHEMISTRY

Name of the Staff	:	**Dr. M. Makeswari and Ms.B. Prabha**
Department	:	**Chemistry**
Title of the Paper	:	**Physical Chemistry Practical-I**
Paper Code	:	**16CHP311**
Class	:	**II-M.Sc-Chemistry**
Year and Semester	:	**II Year and III-Semester**
Total Hours	:	**48 Hours**

S.No	Duration Hours	Name of the Experiments	Support materials
1	**4**	Determination of molecular weight –Rast's micro method	**R1**
2	4	Freezing point depression-Eutectic system	**R1**
3	4	Heat of solution from solubility	**R1**
4	4	Determination of Distribution coefficient of I_2 between CCl_4 & H_2O	**R1**
5	4	Determination of Equilibrium constant	**R1**
6	4	Determination of cell constant and verification of Debye-Huckel Onsager law	**R1**
7	4	Verification of Ostwald dilution and Kolhrawch's Law	**R1**
8	4	Conductometric determination of pK_a of a weak acid	**R1**
9	4	Conductometric Titration of Strong acid Vs Strong Base	**R1**
10	4	Conductometric Titration of Weak acid Vs Weak Base	**R1**
11	4	Viva -voce	

12	4	Model practical Exam	

REFERENCE BOOKS

1. **R1:** Practical chemistry by A.O.Thomas, 2003, Scientific Book centre.

PHYSICAL CHEMISTRY PRACTICAL-I

LAB MANUAL

FOR

II M.Sc., CHEMISTRY STUDENTS

DEPARTMENT OF CHEMISTRY

KARPAGAM ACADEMY OF HIGHER EDUCATION

(Deemed to be University)

(Established Under section 3 of UGC Act, 1956)

Eachanari Post, Pollachi main road,

Coimbatore-641021

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CONTENS

Ex.NO	NAME OF THE EXPERIMENT	Page No
1	Determination cell constant	3
2	Verification of Debye Huckel equation	5
3	Dissociation Constant of a weak electrolyte	8
4	Dissociation of Verification of Kolhrawch's Law	11
5	Titration of Strong acid Vs Strong Base	14
6	Eutetic System	17
7	Rast micro method	20
8	Distribution of coefficient of I_2 in CCl_4 & H_2O	22
9	Determination of Equilibrium constant	26
10	Determination of KI by partition method	31

CONDUCTOMETRIC TITRATIONS

Experiment No 1

DETERMINATION OF CELL CONSTANT OF THE GIVEN CONDUCTANCE CELL

Aim

To determine the cell constant of the given conductance cell

.

Principle

The resistance of any conductor varies directly at its length (*l cm*) and inversely as its area [*a* sq.cm]; that is

$$R = \rho . l/a \ldots\ldots\ldots\ldots\ldots\ldots\ldots(1)$$

Where, ρ is a constant, called the specific resistance of the conducting material. '*l*' is the distance of separation of electrodes, 'a' is the area of cross section. The reciprocal of ρ which is 'k' is the specific conductance

$$= \frac{1}{k} \times l/a \qquad \ldots\ldots\ldots\ldots\ldots\ldots(2)$$

$$l/a = R.k \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(3)$$

l/a is called cell constant and this may be obtained by direct measurement. But this rarely done since it is possible to measure it by means of a solution of known specific conductance, KCl solutions are used invariably, for this purpose for they have been measured with great accuracy in cells of known dimension

Materials Required

Digital conductivity meter, conductivity cell, conductivity water, 1 N KCl solution.

Procedure

Exactly 0.1N and 0.02N solution of KCl were prepared 40 ml of 0.1N KCl was taken in a clean beaker, dipped the conductivity cell and the connection were made and the conductance was found. The experiment was repeated with 0.02N KCl solution and from the conductance and the specific conductance of two solutions the cell constant was calculated.

Observations and calculations

Concentration of KCl in N	Observed conductance in(ohm^{-1}) C = 1/R	Specific conductance at 25 ^{0} C (ohm-^{1} cm^{-1}) K	Cell constant K= k /c cm^{-1}
1.0		0.11173	
0.1		0.012886	
0.01		0.0014114	

Result

The cell constant of the given conductivity cell is found to be =------- cm^{-1}

Note.

1. Potassium chloride, 74.6 g in 1 liter of distilled water = 1N
2. Conductivity water, this is obtained by redistilling distilled water with addition of a little potassium permanganate and traces of caustic soda in an all glass apparatus. The specific conductivity of water should be less than 5X 10^{-6} mhos cm^{-1}

Concentration of KCl in N	Specific conductance at 0 ^{0} C (ohm^{-1} cm^{-1}) k	Specific conductance at 18^0 C (ohm-^{1} cm^{-1}) k	Specific conductance at 25^0 C (ohm-^{1} cm^{-1}) k ^{1}
1.0	0.06543	0.09820	0.11173
0.1	0.007154	0.011192	0.012886
0.01	0.0007751	0.0012227	0.0014114

Viva questions

1. Why the equivalent mass of a potassium chloride is its molecular mass itself?
2. What are the classes of conductors?
3. Define metallic conductance
4. Define electrolytic conductance
5. Define transport of matter
6. What is the Faraday Laws of electrolysis?
7. Define specific and equivalent conductance
8. What is the relation between specific and equivalent conductance?
9. Define ionic mobility
10. What is Electro chemistry?

Experiment No 2.

DETERMININATION OF EQUIVALENT CONDUCTANCE OF A STRONG ELECTROLYTE OF DIFFERENT CONCENTRATIONS AND HENCE TO TEST THE VALIDITY OF THE DE-BYE HUCKEL ONSAGER EQUATION

Aim

To determine the equivalent conductance of a strong electrolyte of different concentrations and hence to test the validity of the De-bye Huckel Onsager equation.

.

Principle

Strong electrolytes are those which ionize completely at all dilutions. Changes in the conductance of electrolyte solution with concentration occur, due to changes both in the increase in the conductance of the solution of strong electrolyte with dilution is mainly due to increase of mobility of the ions.

The Debye-Hukel Onsager equation may be written as

$$\lambda = \lambda_0 - (A - B\lambda_0)\sqrt{C}$$

Where ‘λ’ is the equivalent conductance at a particular concentration ‘c’. λ_0 is that indefinite dilution and A and B are constants for an electrolyte of a given valency type in a particular solvent at a given temperature by the extrapolation of the plot of λ against $\sqrt{c}$ to zero concentration, λ_0 can be calculated.

Materials Required

Digital conductivity meter, conductivity cell, conductivity water, 1 N HCl solution, standard KCl solution for determination of cell constant.

Procedure

Exactly 0.2N solution of strong electrolyte solution was prepared. A conductivity cell whose cell constant was known was dipped inside the solution for which the conductance was to be measured. Then different concentrations of the strong electrolyte prepared from 0.2Nsolution by dilution and their conductance was measured. The corresponding λ_0 was plotted against $\sqrt{c}$ at each concentration by extrapolation of the straight line λ0 was calculated from the slope of the line using the expression.

$$\lambda = {}_0 - (A - B\lambda_0)\sqrt{C}$$

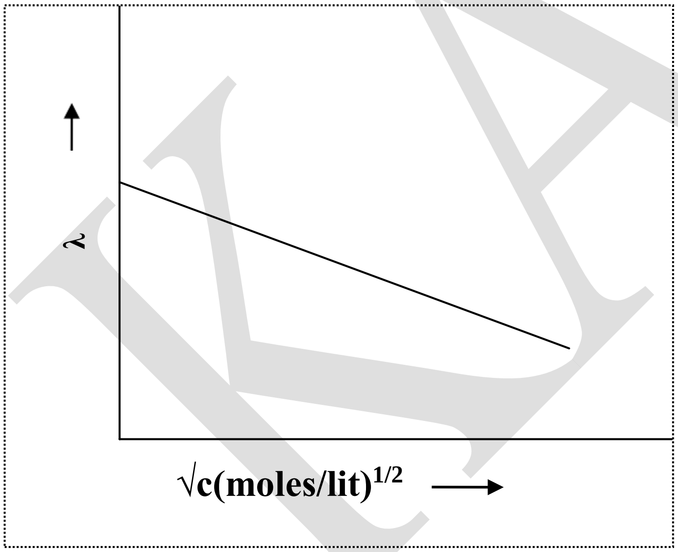
And assuming the values of A and B to be 60.2 and 0.229 respectively (aqueous solution at 25^0C).

Observations and calculations

Cell constant (x) =-------- cm^{-1}

Concentration of HCl (c) moles/lit	Observed conductance in (ohm^{-1}) C = 1/R	Specific conductance a (ohm-^{1} cm^{-1}) K = x X C	λc =K/c Ohm^{-1} cm^2/gm equ	$\sqrt{c}$ (moles/lit)$^{1/2}$

Graph



Result

The equivalent conductance at infinite dilution of the given strong electrolyte is found to be

(i) From slope = ------------ohm^{-1} cm^2/gm equ.

(ii) From y intercept = ----------ohm^{-1} cm^2/gm equ.

Note.

1. Hydro chloric acid, 86 ml in 1 liter of distilled water = 1N
2. Various concentration range of HCl, 0.01 N to 0.2 N
3. The increase of the equivalent conductance of solutions of strong electrolytes with dilution is mainly due to increased mobility of the ions.
4. For strong electrolyte instead of HCl, sodium chloride is also used.
5. Debye – Huckel - Onsager treatment cannot be applied to weak electrolytes.

Viva questions

1. What are the differences between strong and weak electrolyte?
2. Define electrolyte
3. Define Ohm’s Law
4. Define degree of dissociation of the electrolyte
5. Define dielectric constant
6. What do you mean by interionic forces?
7. Define ionic doublets
8. Define asymmetric effect
9. What is electrophoric effect?
10. Give the example for uni-univalent electrolytes.

Experiment No 3.

DETERMINATION OF DISSOCIATION CONSTANT OF THE GIVEN WEAK ELECTROLYTE USING OSTWALD'S DILUTION LAW AND HENCE THE PKA VALUE OF WEAK ELECTROLYTE

Aim

To determine the dissociation constant of the given weak electrolyte using Ostwald's dilution law and hence the pka value of weak electrolyte.

Principle

Weak electrolytes are those which dissociate only slightly even in very dilute solutions. The increase in equivalent conductance is not primarily due to the mobility of ions, but it is largely due to an increase in the number of ions with dilution. According to Arrhenius theory, the dissociation constant of a weak electrolyte is not complete even in dilute solutions and degree of dissociation 'α'at any concentration is given by the relation

$$\alpha = \frac{\lambda_C}{\lambda_\infty}$$

λ_∞ can be obtained from the known conductance of constituent ions. The dissociation constant ka of weak electrolyte can be calculated using the relation

$$K_a = \frac{\alpha^2 C}{1-\alpha}$$

Where 'c' is the concentration in gm mol / liter

Materials Required

Digital conductivity meter, conductivity cell, conductivity water, 1 N acetic acid solution, standard KCl solution for determination of cell constant.

Procedure

0.2N, 0.15N, 0.1N, 0.05N, 0.025 N………..0.0015N solution of weak acid were prepared using conductivity water. Using conductivity cell of known cell constant, the equivalent conductance of all these solutions were determined. Then, the dissociation constant were calculated using Ostwald's dilution law. From the value of dissociation constant, Ka of the weak acid, Pka value was calculated using the expression

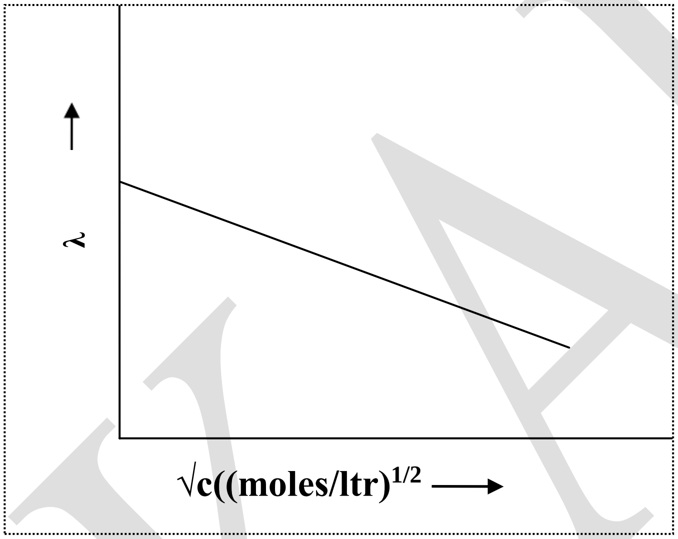
PKa = - log Ka

Observations and calculations

Cell constant (x) =-------- cm^{-1}

Concentration of weak electrolyte (c) Moles/lit	Observed conductance in(ohm^{-1}) C = 1/R	Specific conductance (ohm^{-1} cm^{-1}) K = x X C	λc =K/c Ohm^{-1} cm^2/gm equ	$\sqrt{c}$ $(moles/lit)^{1/2}$	$\alpha = \frac{\lambda c}{\lambda \alpha}$	$Ka=\alpha^2 c/1- \alpha$

Graph



Result

The dissociation constant of the weak acid is found to be

i. Ka =

ii. Pka=

Note.

1. Acetic acid, 58 ml in 1 liter of distilled water = 1N
2. For weak electrolyte instead of CH_3COOH , NH_4OH is also used.
3. Debye – Huckel - Onsager treatment cannot be applied to weak electolytes.

Viva questions

1. What are the differences between strong and weak electrolyte?
2. Define electrolyte
3. Define Ohm’s Law
4. Define degree of dissociation of the electrolyte
5. Define dissociation constant
6. What are the limitation of Arrhenius theory
7. Define ionic doublets
8. Give the example for uni-univalent, bi-univalent and bi-bivalent electrolytes.

Experiment No. 4

DETERMININATION OF EQUIVALENT CONDUCTANCE OF A WEAK ELECTROLYTE AT INFINITE DILUTION USING KOHLRAUSH'S LAW.

Aim

To determine the equivalent conductance of a weak electrolyte at infinite dilution using Kohlraush's law.

Principle

λ_0 for weak electrolyte, like acetic acid cannot be obtained by the method of extrapolation like in strong electrolyte (Debye – Huckel - Onsager treatment), λ_0 greatly increases at low dilution. It can be calculated using Kohlraush's law of independent migrations of ions, according to which the equivalent conductance at infinite dilution is the sum of ionic conductance of the ion constituting the electrolyte. Thus,

$$\lambda_0 (CH_3COOH) = \lambda_0 (CH_3COONa) + \lambda_0 (Hcl) - \lambda 0 Nacl$$

It is thus possible to evaluate λ_0 for acetic acid from λ_0 value for sodium acetate, hydrochloric acid and sodium chloride. Since all these are strong electrolytes, λ_0 can be determined by the method of extrapolation of the plot of λc against $\sqrt{c}$.

Materials Required

Digital conductivity meter, conductivity cell, conductivity water, 1 N acetic acid, 1 N sodium chloride, 1 N hydro acid, standard KCl solution for determination of cell constant.

Procedure

Exactly 0.2N solution of strong electrolyte such as hydrochloric acid was prepared. A convenient amount of the solution was taken in a dry beaker and the conductivity cell of known cell constant was dipped in it. The conductance of the solution was measured. Then 0.18N, 0.16N, 0.14N, 0.2N solution of HCl were prepared and thus conductances were measured in each case. The corresponding equivalent conductance λc was calculated. λc was plotted against $\sqrt{c}$ at each concentrated and by extrapolation of the line λ_0 was determined. The same procedure was followed for determining λ_0 values of sodium acetate and sodium chloride using the formula.

$$\lambda_0 CH_3COOH = \lambda_0 (CH_3COONa) + \lambda_0 (HCl) + \lambda_0 (NaCl)$$

λ_0 for acetic acid was calculated.

Observations and calculations

Cell constant (x) =-------- cm^{-1}

λ_0 (CH_3COONa)

Concentration of CH_3COONa (c) moles/lit	Observed conductance in(ohm^{-1}) C = 1/R	Specific conductance (ohm-^{1} cm^{-1}) K = x X C	λc =K/c Ohm^{-1} cm^2/gm equ	$\sqrt{c}$ (moles/lit)$^{1/2}$

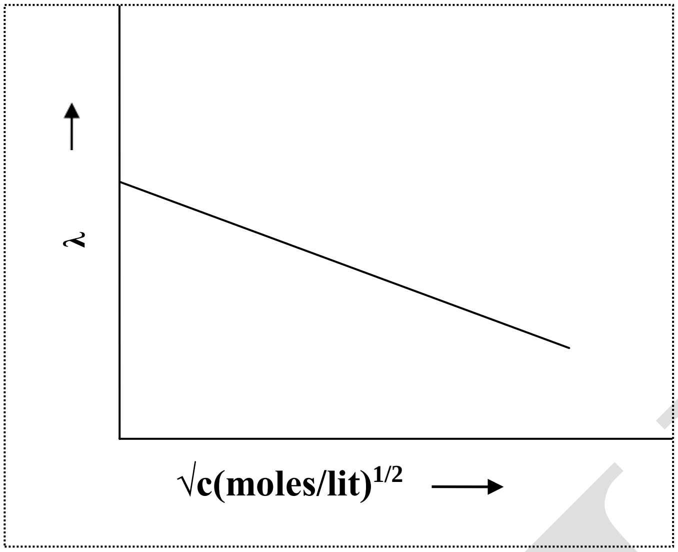
λ_0 (HCl)

Concentration of HCl (c) moles/lit	Observed conductance in(ohm^{-1}) C = 1/R	Specific conductance (ohm-^{1} cm^{-1}) K = x X C	λc =K/c Ohm^{-1} cm^2/gm equ	$\sqrt{c}$ (moles/lit)$^{1/2}$

λ_0 (NaCl)

Concentration of NaCl (c) moles/lit	Observed conductance in(ohm^{-1}) C = 1/R	Specific conductance (ohm-^{1} cm^{-1}) K = x X C	λc =K/c Ohm^{-1} cm^2/gm equ	$\sqrt{c}$ (moles/lit)$^{1/2}$

Graph



Result

The equivalent conductance at infinite dilution of acetic acid is found to be = ---- ohm^{-1} cm^{2} /g eq

Note.

1. Sodium acetate, 82.03g in 1 liter of distilled water = 1N
2. HCl, 86 ml in 1 liter of distilled water = 1N
3. NaCl, 58.45g in 1 liter of distilled water = 1N

Viva questions

1. What are the differences between strong and weak electrolyte?
2. Define transport number
3. What is the basic principle of Kohlraush's law?
4. Define degree of dissociation of the electrolyte
5. Define dissociation constant
6. What are the limitations of Arrhenius theory?
7. Define infinite dilution of electrolyte
8. Give the example for uni-univalent, bi-univalent and bi-bivalent electrolytes.

CONDUCTOMETRIC EXPERIMENTS

Experiment No.5

DETERMININATION OF AMOUNT OF HYDROCHLORIC ACID PRESENT IN THE GIVEN SOLUTION BY CONDUCTIVITY METHOD

Aim

To determine the amount of hydrochloric acid present in the given solution by conductivity method.

Principle

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. So the mobility of the hydrogen ions will be greater than the sodium ions so the conductance of the solution containing sodium ions will be less than the solution containing hydrogen ions. Same principal is used here in this experiment ,initially solution contain the hydrogen ions when this solution is titrated against the base solution ,the base reacts with the acid and the number of the hydrogen ions go on decreasing. Finally a stage reaches when there is no hydrogen ion in the solution so the conductivity decreased and remains constant. If further base is added then the number of negative ions increases and the conductivity go on increasing. The point at which the conductivity becomes constant is the equivalence point. The volume of base used at equivalence point is used to calculate the molarity of acid and then the strength of acid. Consider a solution of a strong acid, hydrochloric acid, HCl for instance, to which a solution of a strong base, sodium hydroxide NaOH, is added. The reaction occurs. For each amount of NaOH added equivalent amount of hydrogen ions is removed. Effectively, the faster moving H^+ cation is replaced by the slower moving Na^+ ion, and the conductivity of the titrated solution as well as the measured conductance of the cell fall. This continues until the equivalence point is reached, at which we have a solution of sodium chloride, NaCl. If more base is added an increase in conductivity or conductance is observed, since more ions are being added and the neutralization reaction no longer removes an appreciable number any of them. Consequently, in the titration of a strong acid with a strong base, the conductance has a minimum at the equivalence point. This minimum can be used instead of an indicator dye to determine the endpoint of the titration. Conductometric titration curve that is a plot of the measured conductance or conductivity values against the number of milliliters of NaOH solution

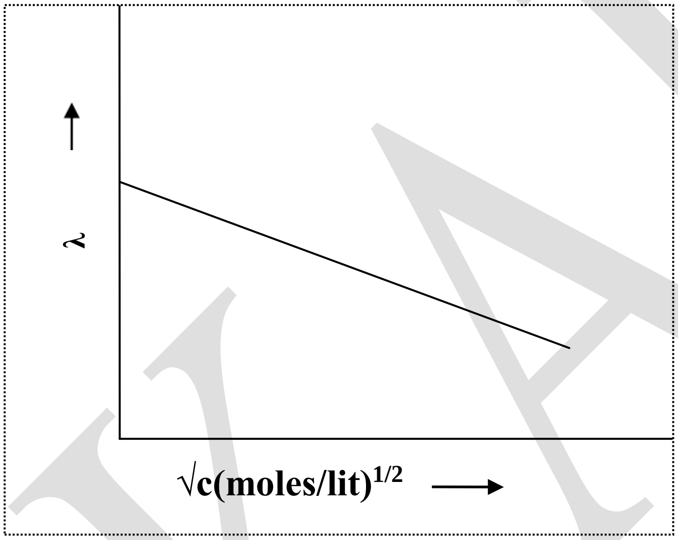
Materials Required

Digital conductivity meter, conductivity cell, conductivity water, standard KCl solution for determination of cell constant, 0.01N HCl, 0.2N NaOH.

Procedure

The given strong acid was made up to exactly 100cc in volumetric flask using conductivity water. 20ml of this made up solution was pipette out after shaking into a clean beaker and added about 20cc of conductivity water. The conductivity cell was dipped into it. The conductance offered by solution for the cell was found out. From the burette sodium hydroxide was added in 1cc portions. In the beginning and in 0.2ml portions near the end point and the corresponding conductance were measured. A graph was drawn by taking v×c along t y axis and colume of sodium hydroxide along x axis. The intersection of the two straight lines gives the end point and from this strength of the given hydrochloric acid present in the whole of the given solution was calculated.

Graph



Observations and calculations

Volume of NaOH in cc	Observed conductance in m-mho	v×c in cc m-mho

Volume of NaOH solution (V_1) = ……ml

Strength of NaOH solution (N_1) = 0.1 N

Volume of strong acid (HCl)(V_2) = 20 ml

Strength of strong acid (HCl) (N_2) = V_1N_1/V_2

= …… N

Amount of strong acid present in the whole of the given solution = $\frac{Normality \times Eq.wt}{10}$

= ....g

Result

(i) The strength of the given acid was found to be = ……N

(ii) The amount of acid present in the whole of the given solution = **……g**

Note

1. Same procedure is adapted for acetic acid estimation.

Viva questions

1. What is strong acid and strong base?
2. What is the relationship of a strong acid and a strong base?
3. Do strong base has a strong conjugate acid?
4. Is KBr a strong acid or strong base?
5. What are the characteristics of a strong base or strong acid?
6. What is alkalinity?
7. Which ions impart alkalinity to the natural water?
8. How conductance is related to the concentration of the ions?
9. Why conductance decreases on addition of NaOH to HCI?

THERMAL EXPERIMENTS

Experiment No 6

DETERMININATION OF EUTECTIC TEMPERATURE AND EUTECTIC COMPOSITION OF A GIVEN SYSTEMS.

Aim

To determine the eutectic temperature and eutectic composition of a given systems.

Principle

When the liquid mixture of two substances A and B which do not form a compound is cooled, solid begins to separate at a definite temperature. This temperature is called the freezing point of the mixture. Adding of anyone of the component say B to A lowers the melting point of substance A.

The depression in melting point. thing point of the pure substance is propotional to the amount of the other substance added. At a particular temperature composition of the mixture of two components A and B have the lowest possible freezing point is known as eutectic temperature is eutectic composition > below the eutectic temperature liquid doesn't exist. Mixture of substance A and B in varing composition are latter the corresponding freezing point of the melts and determined.

Melting point of pure A and B are also found out a graph is drawn with composition of the mixture against freezing temperature are read from the graph.

Materials Required

Glass specimen tube, sensitive thermometer, air jacket, biphenyl amine, naphthalene

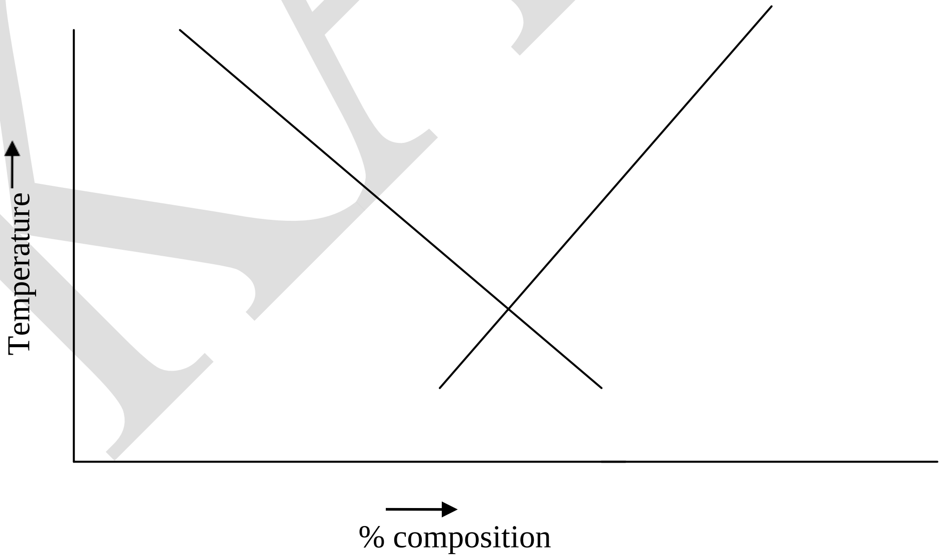
Procedure

The glasss specimen tube was cleaned well and dried. It is fitted with two holed rubber cork through which a sensitive thermometer and a stirrer was passed. This was fitted into an outer tube calle. an air jacket.

4g of the soid was weighed accurate and introduced into the specimen tube fitted with a cork carrying a thermometer and stirrer . it was then fixed into the air jacket and immersed in a waterbath and heated. The melting point of pure ‘A’ was noted. It was further heated till it melts completely. Then the apparatus was taken out of the hot water bath and allow to cool with constant stirring . the temperature at which first solid appeared was noted. The mean value of two temperature readings were taken at the freezing point of pure ‘A’

0.4g of the substance ‘B ; was accurately weighed and carefully transferred into the specimen tube. It was heated in the hot liquid bath till it become a homogenous liquid. The freezing point of the mixture was noted. The experiments was repeated several times by adding known amount of”B” to ‘A’ and recorded . the experiments was repeated by taking 4g of pure ‘B’ and addition ofg of portion ‘A’ to this several times. The melting point of pure ‘B’ and the freezing point of the mixture of various composition of A and B were the determined.

A graph was plotted by taking composition along x axis and the freezing point of mixture along y axis. Two curve were obtained. One showing the change in freezing point ar ‘A’ due to the addition of ‘B’ and the other showing the change in freezing point of ‘B’ due to addition of ‘A’. the point at which two curves meet gives the eutectic temperature of the binary system ‘A’ and ‘B’. the eutectic composition was obtained by dropping perpendicular as line to the composition axis and the eutectic temperature by dropping a perpendicular line to the temperature axis from eutectic point.



Observations and calculations

Weightof A in gms	Weight of B in gms	% of A $A/A+B$	% of B	Melting point ^{0}C	Freezing point^{0}C	Mean temp ^{0}C

Result

1. Melting point of A =
2. Melting point of B =
3. Eutectic composition of A=
4. Eutectic composition of B =
5. Eutectic temperature=

Viva questions

1.Define eutectic temperature.?

2.Define eutectic composition.?

3. What are the application of eutectic system.?

4.Define mullite.?

5.What is collective properties.?

6. Define elevation of boiling point.?

Experiment No 7

DETERMINATION OF MOLECULAR MASS BY RAST'S METHOD

Aim

To determine the Molecular mass by Rast's method

Principle

Presence of dissolved non-volatile substances depress the melting point of camphor.

The depression in melting point.

$$\Delta T = K \times \frac{1000 \times W2}{M2 \times W1}$$

Where K= molal depression constant
W1 = mass of solvent
W2= mass of solute
M2= molecular mass of solute

The molal depression constant is the depression of the freezing point of a solvent when one gram molecular mass of a solute is dissolved in 100g of the solvent. This method can be used only for non-volatile solutes which dissolve in molten camphor without reaction with it.

Materials Required

Camphor, acetanilide, glycerine bath, thermometer, flat bottom test tube,

Procedure

Determination of K for camphor using reference solute

An ignition tube is weighed accurately. About one gram of pure camphor is taken and again weighed. About 0.1g of acetanilide is introduced and gram weighed.

The tube is corked and heated on a glycerine bath. When it is melted to become a homogeneous solution, it is poured on a clean porcelain dish. When it is solidified, it is powdered and its melting point is determined by calillary method. Similarly the melting of pure camphor is also determined. The difference between the two values gives the depression in the melting point , ΔT.

Observations and calculations

Mass of empty tube = ag

Mass of empty tube + camphor =bg

Mass of camphor = (b-a)= W1 g

Mass of tube+ camphor+ acetanilide = cg

Mass of acetanilide = (c-b)=W2 g

Melting point of champhor =T_0

Melting point of mixture =T

Depression in melting point =T_0-T= ΔT

$$\Delta T = \frac{1000 \times K\, X W2}{M2\, X W1}$$

The same experiment is repeated with the given solute and the depression in melting point is noted. Knowing the values of K and ΔT, molecular mass of solute is calculated from the following equation.

$$M2 = \frac{1000\, X\, KX\, W2}{W1\, X\, \Delta T}$$

Result

The molecular weight of given solute = g

Viva questions

1. Define molecular mass and equivalent mass.?

2.Define molal depression constant.?

3.Differenciate volatile and nonvolatile solute.?

4.Define elevation of boiling point.?

6.What is cryoscopic constant .?

7.Why the solvent melting point is decrease or increase with solvent.?

8. What is the proposed reaction for camphor and acetanilide.?

PARTITION EXPERIMENTS

Experiment No 8

DETERMINATION OF PARTITION COEFFICIENT OF IODINE BETWEEN CARBON TETRA CHLORIDE AND WATER

Aim

To determine the partition coefficient of iodine between carbon tetra chloride and water.

Principle

When a solute is shaken up with two immiscible solvents, if it is soluble in each of them it will distributed itself between the two solvents. In accordance with the distribution law. If the solute is neither associated nor dissociated both the solvents the partition will take up accordance to the following equation called Nernst distribution law

$$k = \frac{c1}{c2}$$

Where c1 ,c2 refers to the concentration of solute in solvents 1 and 2 respectively and k is partition co efficient or distribution co efficient iodine distributes itself in a definite manner between carbon tetra chloride and aquous layer at constant temperature

Then $\frac{c1}{c2}$ =constant.

Procedure:

Saturated solution of iodine in CCL_4 and water are measured out in two leak proof bottles as follows.

B.NO	Volume of iodine In CCL_4 in cc	Volume of CCL_4 in cc	Volume of water in cc
I	15	5	100
II	10	10	100

The bottles are stoppered and carefully shaken vigorously for about an hour. The bottles were then placed in a thermostat to attain equilibrium at constant temperature. At frequent intervals removed the bottles one by one and shook vigorously for about one or two minutes and the replaced them in a thermostat. After the attainment of equilibrium pipetted out 20ml of aqueous layer into a conical flask. Added 1g of KI ans titrated against $N/100$ with thio solution using starch as an indicator. The disappearance of blue colour marked the end point repeated the titrated to get concordant values.

In a similar manner pipetted out 5ml of the organic layer into a conical flask and added 1g of KI and titrated against $N/10$ thio solution using starch as an indicator . the same procedure was repeated with the solution in other bath knowing the concentration of iodine in organic layer and aqueous layer, the partition co-efficient was calculated.

Observations and calculation

Normality of potassium dichromate = $\frac{wt}{lit}/eq.wt$

Standardization of sodium thio sulphate

Titration of standard potassium di chromate against sodium thiosulphate

Volume of potassium dichromate Incc	Burette reading(ml)		Volume of sodium thio sulphate	Concordant value
	Initial	Final		

Volume of potassium dichromate (V_1) =

Normality of potassium dichromate (N_1 =

Volume of sodium thiosulphate(V_2) =

Normality of sodium thio sulphate ($N_{2)}$ =

Distribution co-efficient of iodine between carbon tetra chloride and water.

Titration of iodine in organic layer against sodium thio sulphate.

Volume of iodine In organic layer	Burette reading		Volume of Thio in cc
	Initial	Final	

Titration of iodine in aqueous layer against sodium thio sulphate

Indicator : starch

Volume of iodine In organic layer	Burette reading		Volume of Thio in cc
	Initial	Final	

Calculations:

Bottle I organic layer:

Volume of sodium thio sulphate (v1) =

Normality of sodium thio sulphate(N1) =

Volume of organic layer pipetted out(V2) =

Normality of iodine in organic layer (N2) =

Concentration of iodine in organic layer c1 =

Aquous layer:

Volume of sodium thio sulphate (v1) =

Normality of sodium thio sulphate(N1) =

Volume of organic layer pipetted out(V2) =

Normality of iodine in organic layer (N2) =

Concentration of iodine in aquous layer c =

$$\text{Partition co-efficient } [K_D] = \frac{\text{Concentration of iodine in organic layer in gm moles /lit}}{\text{Concentration of iodine in aquous layer gm moles/li}}$$

$$K_D = \; = \frac{c1}{c2}$$

Result:

Distribution co-efficient of iodine between CCL_4 and water K_D =

Viva questions

1. Define Nernst distribution law.?
2. Define distribution co-efficient.?
3. Differentiate organic and aqueous layer.?
4. Give two examples are non miscible solvent.?
5. Define equilibrium constant.?
6. When association occurs in one solvent.?
7. Define Henry's law of distribution .?
8. Differentiate single and multiple extraction.?
9. What is the application of partition experiments

Experiment No 9

DETERMINATION OF EQUILIBRIUM CONSTANT BY DISTRIBUTION METHOD

Aim

To determination of equilibrium constant by distribution method

To determine the equilibrium constant for the reaction

$KI + I_2 \rightarrow KI_3$ by distribution method

Principle

The partition law is obeyed Well for the distribution of iodine between CCL_4 andwater. At constant temperature the partition co-efficient is given by

$$KD = \frac{concentration\ of\ iodine\ in\ organic\ layer\ in\ gm\ moles\ /lit}{concentration\ of\ iodine\ in\ aqueous\ layer\ gm\ moles\ /lit}$$

The law doesn't hold good in the distribution of iodine between organic and aqueous layer of KI because in aqueous layer the complex is formed. The facture of distribution law is only only apparent and it is expected to hold good provided the concentration of iodine in aqueous layer is taken into a consideration of iodine is distributed between CCl_4 and aqueous solution of KI, the total concentration of iodine in aqueous layer is given by[concentration of iodine in free+ concentration of I_3] which can be determined by titration with standard thio solution. The concentration of iodine in organic layer can also be determined by titration with standard thio solution. Then the concentration of free iodione in aquous layer can be determined by dividing the concentration of iodine in organic layer by partition co-efficient. It is used to calculate the vales of Cl_3^- and $CI^{-.}$ If the initial concentration of KI is known and its Cl_3^- , CI_2, $CI^{-.}$ Are un known the equilibrium constant for the reaction.

$$KI + I_2 \rightarrow KI_3$$

$$kc = \frac{I^{3-}}{[KI][I^{2-}]}$$

Let the concentration of iodine in organic layer be c1 gm moles/litre concentration of iodine in aqueous layer be c2 gm moles/lit.

Concentration of free iodine in aquous layer =$\frac{c1}{KD}$ gm moles/ lit

Concentration of I_3^- =$c2 - \frac{c1}{KD}$

Let the initial concentration of I_3 be gm moles /lit since the mole of I_3^- formed , out of one mole of I^- concentration of free iodine at equilibrium

$$= 0.1\text{-}\ c2 - \frac{c1}{KD}$$

$$= \frac{\frac{c2-(\frac{c1}{KD})}{c1}}{K[0.1(I3)]}$$

Procedure

Two leak proof stoppered bottles were taken and made the following mixture, stoppered the bottles shaken well and kept in a thermostat attain equilibrium.

B.NO	Volume of iodine In CCL_4 in cc	Volume of CCL_4 in cc	Vo;ume of0.1 N KI solution in cc
I	20	0	100
II	15	5	100

Pipetted out 20ml of aqueous layer into a conical flask containing concentration of 5% K solution and titrated against N/10 thio solution using starch as indicator. Similarly pipetted out 5ml of organic layer into a conical flask containing 10ml of 5% KI and titrated against N/10 thio solution using starch as indicator . repeated the same procedure for the other bottle from the data equilibrium constant can be calculated.

Observations and calculation

Determination of equilibrium constant by distribution method

Normality of potassium dichromate = $\frac{wt/lit}{eq.wt}$

Standardization of sodium thio sullphate

Titration of standard potassium di chromate against sodium thiosulphate

Indicator : strach

Volume of potassium dichromate Incc	Burette reading(ml)		Volume of sodium thio sulphate	Concordant value
	Initial	Final		

Volume of potassium dichromate (V_1) =

Normarlity of potassium dichromate (N_1 =

Volume of sodium thiosulphate(V_2) =

Normality of sodium thio sulphate ($N_{2)}$ =

Titration of iodine in organic layer against sodium thio sulphate.

Volume of iodine In organic layer	Burette reading		Volume of Thio in cc
	Initial	Final	

Titration of iodine in aquous layer againt sodium thio sulphate

Indicator : starch

Volume of iodine In organic layer	Burette reading		Volume of Thio in cc
	Initial	Final	

Calculations:

Bottle I organic layer:

Volume of sodium thio sulphate (v1) =

Normality of sodium thio sulphate(N1) =

Volume of organic layer pipetted out(V2) =

Normality of iodine in organic layer (N2) $=v1 \times \frac{N1}{N2}$

Concentration of iodine in organic layer c1 =

Aquous layer:

Volume of sodium thio sulphate (v1) =

Normality of sodium thio sulphate(N1) =

Volume of organic layer pipetted out(V2) =

Normality of iodine in organic layer (N2) $= v1 \times \frac{N1}{N2}$

Concentration of iodine in aquous layer c2 =

Concentration of iodine in aquous layer c3= $\frac{c1}{KD}$

Concentration of KI3 (c2-C3) = C4---gmmol/lit

Concentration of KI used 0.1mol/lit

Concentration of KI at equilibrium =0.1- c4 ---------- gmmol/lit

Eauilibrium constant $\frac{[KI_3]}{[KI]\,[I_2]}$ =

Result

The equilibrium constant for the tri iodide formation was found to be

(i)

(ii)

Experiment No 10

DETERMININATION OF CONCENTRATION OF THE GIVEN KI SOLUTION USING THE VALUES OF EQUILIBRIUM CONSTANT AT THE REACTION

Aim

To determine the concentration of the given KI solution using the values of equilibrium constant at the reaction

$$KI + I \rightarrow KI_3$$

Procedure

The given KI solution was made up to 250ml. Two stoppered bottles were taken and made the following mixture.

B.NO	Volume of iodine In CCL_4 in cc	Volume of CCL_4 in cc	Volume of given KI solution in cc
I	15	5	100
II	10	10	100

The bottle were stoppered and shaken well for one hour. Bottles were then kept in a thermostat and allowed the layer to separate distinctly. 20ml of the aquous layer and 5ml of the organic layer was pipette out separately from bottle I and II titrated against N/10 sodium thio sulphate solution using starch as indicator the same procedure was repeated with bottle I knowing the equilibrium constant and partition co-efficient of iodine between water and CCl_4 . the strength of the given KI solution was calculated.

Observations and calculation

Normality of potassium dichromate = $\frac{wt/lit}{eq.wt}$

Standardization of sodium thio sullphate

Titration of standard potassium di chromate against sodium thiosulphate

Indicator : strach

Volume of potassium dichromate Incc	Burette reading(ml)		Volume of sodium thio sulphate	Concordant value
	Initial	Final		

Volume of potassium dichromate (V_1) =

Normarlity of potassium dichromate (N_1 =

Volume of sodium thiosulphate(V_2) =

Normality of sodium thio sulphate ($N_{2)}$ =

Titration of iodine in organic layer against sodium thio sulphate.

Volume of iodine In organic layer	Burette reading		Volume of Thio in cc
	Initial	Final	

Titration of iodine in aqueous layer against sodium thio sulphate

Indicator : starch

Volume of iodine In organic layer	Burette reading		Volume of Thio in cc
	Initial	Final	

Calculations:

Bottle I organic layer:

Volume of sodium thio sulphate (v1) =

Normality of sodium thio sulphate(N1) =

Volume of organic layer pipette out(V2) =

Normality of iodine in organic layer (N2) $=v1 \times \frac{N1}{N2}$

Concentration of iodine in organic layer c1 =

Aqueous layer:

Volume of sodium thio sulphate (v1) =

Normality of sodium thio sulphate (N1) =

Volume of organic layer pipette out(V2) =

Normality of iodine in organic layer (N2) $= v1 \times \frac{N1}{N2}$

Concentration of iodine in aqueous layer c2 =

Concentration of iodine in aqueous layer c3= $\frac{c1}{KD}$

Concentration of KI3 (c2-C3) = C4---gmmol/lit

Concentration of KI used = x gm mol/lit

Concentration of KI at equilibrium c5 –x-c4

$$\mathrm{K}_{equ} = \frac{[KI3]}{[KI][I2]}$$

$$= \frac{C4}{[x-c4][c3]}$$

$$= \frac{c4}{c3Keq} + c4$$

Result

Concentration of the given potassium isdide solution is

(i) Bottle I =

(ii) Bottle II=