KONGUNADU ARTS AND SCIENCE COLLEGE

(AUTONOMOUS)
Re-accredited by NAAC with A+ Grade - 4th cycle, College of Excellence - UGC
COIMBATORE -29.

DEPARTMENT OF CHEMISTRY

Supported by : DBT STAR College Scheme

LAB MANUAL & SOP
(STANDARD OPERATING PROCEDURE)

CORE CHEMISTRY PRACTICAL – IV
PHYSICAL CHEMISTRY
EXPERIMENTS

Name : ……………………………………………………….

Class : ……………………………………………………….

Roll No. :……………………………………………………….

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Time: 3 hours **Max. Marks : 60**

Distribution of Physical Practical Marks-60

Record	10
Experiment	50

Distribution of Marks for each Experiment

1. Equilibrium Constant

For carrying out the experiment - 13
Remaining Marks given below - 37
Calculation of [KI] - 8
Calculation of $[I_2]$ - 8
Calculation of $[KI_3]$ - 8
Value of equilibrium
Constant error up to 10% - 13
10-12% - 8
12-14% - 5
>14% - No Marks

2. Strength of KI Solution may be given in the range from 0.02N to 0.06N

Calculation of [KI] - 8
Calculation of $[I_2]$ - 8
Calculation of $[KI_3]$ - 8
Calculation
Strength of given [KI] Solution - 5

For wrong calculation of above value, 50% of marks to be deducted for those steps only.

3.HCl or H_2SO_4, 0.5N to be given. If the order of difference between theoretical and candidates value is

Below a factor of 10 Between 10-20 Above 20-37
- reduce 3 marks for each factor -5 marks

4. Critical Solution Temperature of Phenol-Water system is $67.0\ ^0c$

Phenol-Water system
Plot of % Phenol Vs Temp. -37
Error up to ±3°c -15
Error up to ±3°c to 15°c - reduce 3 marks for each percent
Above ±15°c -5

5. Candidate may be instructed to use solutions of strength 1% and below. Unknown solution must be below 0.8%

Plot of %NaCl Vs Temp

Contents

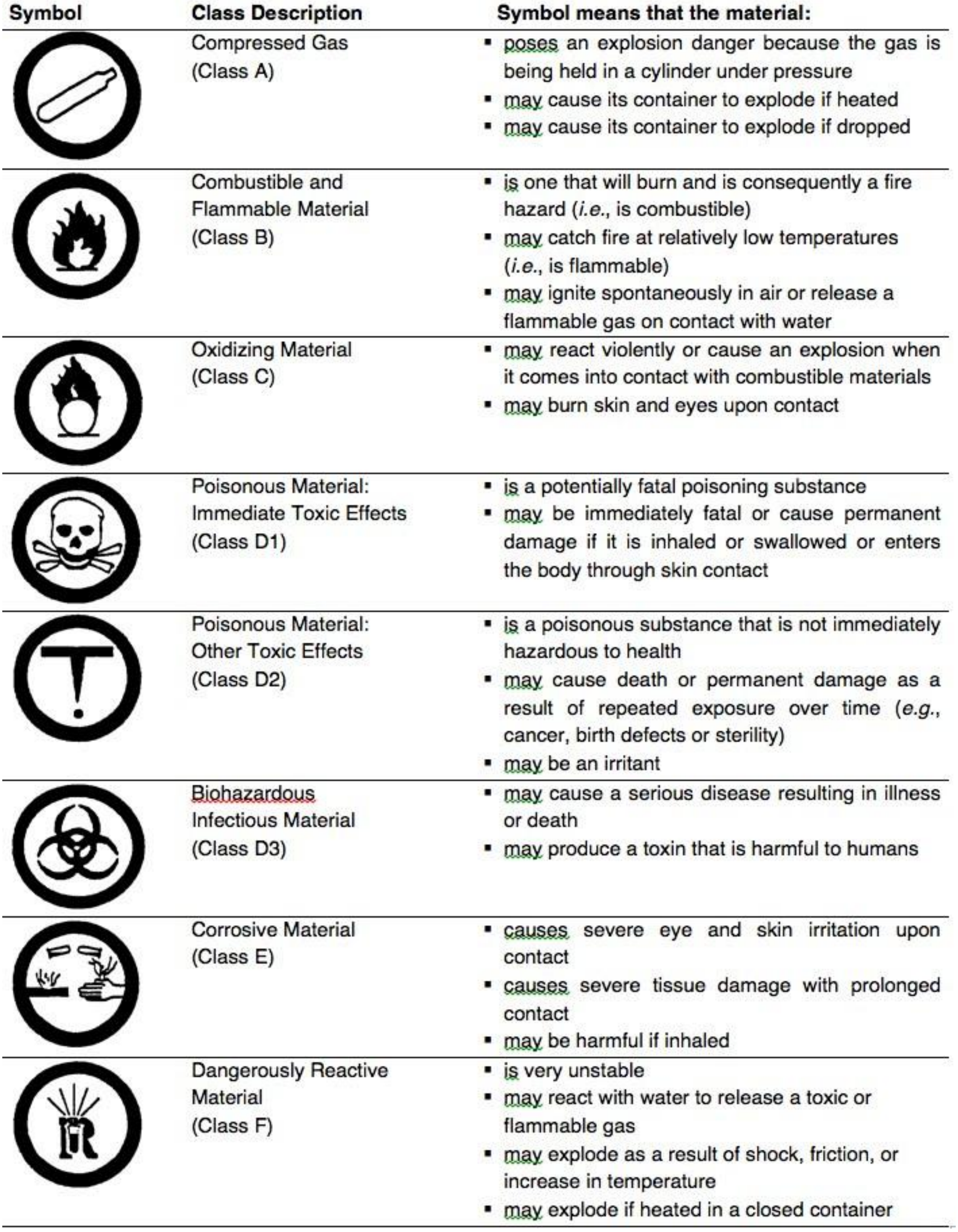
Safety Rules

1. Make sure you are familiar with all the safety information given to you about each experiment before starting the experiment. This includes your manual, these safety guidelines, any posted information or any other information provided to by your Staff or Professor.
2. **Always** wear safety glasses (including during check-in and check-out), except when their removal has been specifically authorized by the Staff or Professor prior to their removal. Contact lenses are forbidden. You must also wear a face shield when requested to by the Staff or Professor.
3. You **must** wear a lab coat (and do it up) in all Chemistry labs.
4. Footwear **must** completely cover the foot and heel (no sandals, baby dolls, ballet flats, mules, open-toed footwear, etc.).
5. You **must** wear long pants (no shorts, capris, skirts, or dresses).
6. Loose hair must be tied back so as to be out of the way. Dangling jewellerymust be removed.
7. Do **not** eat or drink in the lab.
8. Visitors are not allowed to be in the lab.
9. Please keep your work area and the common work areas tidy. Also, please make sure the aisles, safety showers, eyewash stations and doorways are unobstructed.
10. Please leave all glassware, equipment, tools, etc. as clean as or cleaner than you found them.
11. Please clean up spills immediately. If the spill is large or is of a hazardous material, inform the Staff or Professor immediately. Use spill mix to absorb solvent or caustic liquids.
12. Please dispose of waste properly and in a timely manner and according to the instructions provided in your lab manual. If you are not sure, please ask your Staff or Professor for the proper method of disposal.
13. Wash your hands before you leave the lab.
14. Do not remove chemicals or equipment from the lab except when required to do so for analysis.
15. Please notify your Staff or Professor of any serious medical conditions.
16. Do not wear earbuds or earphones while in the lab.

Chemical Safety

1. The vapours of many organic solvents are flammable or combustible. Do not expose electric sparks, open flames and heating elements to organic solvent vapours. UNLESS OTHERWISE STATED, ASSUME ALL ORGANIC SOLVENTS ARE FLAMMABLE.
2. Many chemicals (solid, liquid or vapour) are poisonous. Do not taste chemicals. If it is necessary to smell a chemical, do so by fanning the vapours towards your nose. Never inhale directly. Avoid inhaling dust or fine powders. Use fume hoods and personal protective equipment when necessary.
3. Be extremely careful when transferring, distilling or refluxing volatile liquids.
4. Do not heat, measure or mix any chemicals in front of your face.
5. Never heat a closed system – it will act as a bomb!
6. Never pour water into concentrated acid. Pour acid slowly into water, stirring constantly. Mixing acid with water is often exothermic.
7. Concentrated acids and bases are stored in the fume hood. Do not carry them to your bench.

Make sure test tubes containing reactions are pointed away from people, especially when they are being heated. Pressurized gas cylinders must only be o



Symbol	Class Description	Symbol means that the material:
	Compressed Gas (Class A)	▪ poses an explosion danger because the gas is being held in a cylinder under pressure ▪ may cause its container to explode if heated ▪ may cause its container to explode if dropped
	Combustible and Flammable Material (Class B)	▪ is one that will burn and is consequently a fire hazard (*i.e.*, is combustible) ▪ may catch fire at relatively low temperatures (*i.e.*, is flammable) ▪ may ignite spontaneously in air or release a flammable gas on contact with water
	Oxidizing Material (Class C)	▪ may react violently or cause an explosion when it comes into contact with combustible materials ▪ may burn skin and eyes upon contact
	Poisonous Material: Immediate Toxic Effects (Class D1)	▪ is a potentially fatal poisoning substance ▪ may be immediately fatal or cause permanent damage if it is inhaled or swallowed or enters the body through skin contact
	Poisonous Material: Other Toxic Effects (Class D2)	▪ is a poisonous substance that is not immediately hazardous to health ▪ may cause death or permanent damage as a result of repeated exposure over time (*e.g.*, cancer, birth defects or sterility) ▪ may be an irritant
	Biohazardous Infectious Material (Class D3)	▪ may cause a serious disease resulting in illness or death ▪ may produce a toxin that is harmful to humans
	Corrosive Material (Class E)	▪ causes severe eye and skin irritation upon contact ▪ causes severe tissue damage with prolonged contact ▪ may be harmful if inhaled
	Dangerously Reactive Material (Class F)	▪ is very unstable ▪ may react with water to release a toxic or flammable gas ▪ may explode as a result of shock, friction, or increase in temperature ▪ may explode if heated in a closed container

First Aid

Burns

Burns represent the most common injury in the chemistry lab. They are generally of either the thermal or chemical type. First aid for surface burns of the thermal type involves immersing the burned part in cool water or applying an ice pack to relieve pain and prevent swelling and blistering. The burn is then covered with a clean, sterile, lint-free dressing. Do not apply lotions, ointments or oily dressings. For more serious burns involving deeper layers of skin and tissue, arrange for immediate medical aid.

To minimize injury due to chemical burns, the chemical must be removed from the skin immediately. Flush liquid chemicals away with water; continue to flush for 20 minutes. Continue first aid as for a thermal burn (preceding paragraph). Medical attention should always be sought in the case of chemical burns, especially as delayed reactions are not uncommon.

Chemicals Spilled on the Skin over a Large Area

Quickly remove all contaminated clothing while using the safety shower to flush the chemical from the skin. Time is of the essence here and there is no place for modesty. Continue to flush the affected area with water for at least 20 minutes. Do not use chemical neutralizers. Treat any chemical burns as outlined in the Burns section above.

Chemicals Spilled on the Skin over a Limited Area

Immediately flush the **affected area with cold water**. Once again, time is of the essence. Do not use chemical neutralizers. Treat any chemical burns as outlined in the Burns section above.

Chemicals Splashed into the Eyes

Immediately flood the eyes with water so as to dilute and eliminate the chemical. Hold the eyelids open to facilitate the process. Flush the eyes for at least 20 minutes. Apply clean dressings over both eyes and arrange for immediate medical aid, regardless of the severity of the injury.

Accidental Ingestion of Chemicals

Contact the Poison Control Centre at 108. Relay the following information: identity of the poison, the quantity taken, the route of entry into the body and the time elapsed since the ingestion. Follow the instructions given for treatment.

ATTENDANCE

S.NO	REG.NO	1	2	3	4	5	6	7	8	9	10
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Solution Preparation

1,2. Rast method

Solvent Kf	**Solute**
1. Naphthalene (6.9◦C)	1. Biphenyl
2. Diphenyl (8-8.4◦C)	2. Naphthalene
3. Diphenyl amine (8.4 – 8.8◦C)	3. Dicholorobenzene

For each step error has to be worked out
Melting point of solvent - 07
Error upto ±10% - 30
Error upto ±20% - 15
Above 20% - 05

3. Critical Solution Temperature of Phenol-Water system is 67.0 ^{0}c

Phenol-Water system
Plot of % Phenol Vs Temp. -37
Error up to ±3°c -15
Error up to ±3°c to 15°c - reduce 3 marks for each percent
Above ±15°c -5

4. Candidate may be instructed to use solutions of strength 1% and below. Unknown solution must be below 0.8%

Plot of %NaCl Vs Temp - 20
Determination of % NaCl Solution - 17
Error up to 1% - 17
Error up to 1-2% - 14
Error up to 2-3% - 10
Error above 3% - 05

5. Phase diagram……...…37

Melting point of A - 06
Melting point of B - 06
Construction of phase diagram - 09
Eutectic temperature - 08
Eutectic composition - 08
Error up to 1% - 08
Error up to 2% - 06
Error up to 3% - 04
Error > 3 % - 0

6. 0.1N Acetic acid and 0.1N KCl 100ml each to be given

Calculation of cell constant - 17
Correct Eq. Conductance of Acetic acid - 17

Error up to 10% - 20
Error up to 10% to 20% - reduce 1 mark for each %.

10. 0.02N HCl may be given

Error up to 5% - 37
Error up to 5-10% - reduce 3 marks for each %
Error up to 10-15% - reduce 3 marks for each %
Error above 5% - 05

11. 0.1N Acetic acid 100ml and 0.1N KCl 100ml to be given

Calculation of cell constant - 13
Corrcet Eq. Conductance of Acetic acid - 13
Calculation of dissociation constant - 10
Reduce marks for errors as in **experiment-6 above**

14,15. Colorimetric Experiments

Error upto 1% - 37
Error upto 2% - 30
Error upto 3% - 20
Error > 3% - 05

Calculation:

Weight of the weighing bottle with solvent :

Weight of the weighing bottle after transferring the solvent :

Weight of the solvent W_1:

Weight of the weighing bottle with Solute :

Weight of the weighing bottle after transferring the solute :

Weight of the solute W_2:

Freezing point of the pure solvent (T_1):

Freezing point of the solution (T_2):

Freezing point of the depression $\Delta T_f = (T_1 - T_2)\ ^{o}C$

Molal Depression Constant of the solvent (Kf) =

Molecular weight of the given Solute $= \frac{Kf\ X\ 1000\ X\ W2}{\Delta Tf\ X\ W1}$

Ex.No:1 DETERMINATION OF MOLECULAR WEIGHT- RAST MACRO METHOD

Aim

To determine the molecular weight of the unknown solute (substance) using the molal depression constant of the solvent.

Materials required:

0.5 gram of unknown solute; solvent

Principle

When a non-volatile non-electrolyte is dissolved in a non-electrolyte, solvent, a decrease in freezing point of the solvent is observed. The depression in freezing point is directly related to the molality of the solution.

$M_2 = K_f \text{ X } 1000 \text{ X } W_2/(\Delta T_f \text{ X } w_1)$

Where,

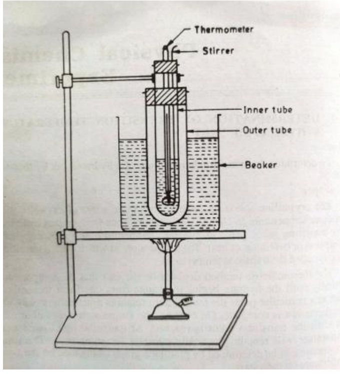
ΔT_f = depression in freezing point

K_f= molal depression constant

M_2 = molecular weight of the solute

W_1 = weight of the solvent

W_2 = weight of the solute



with the value of $K_f$the molecularweight of the unknown solute is determined

Procedure

Weighed quantity of the given solvent was taken in a specimen tube surrounded by an air jacket. This was kept in a water bath and allowed to melt completely. The molten liquid was then allowed to cool with constant stirring. The freezing point was noted then weighed about 0.5 g of the unknown solute accurately and added to the solvent liquid become homogeneous. It was allowed to freeze with constant stirring and freezing point of the mixture was noted. The difference between the freezing point of the solvent and the mixture gives depression constant. Knowing K_f,the molecular weight of the given solute was calculated.

DETERMINATION OF MOLECULAR WEIGHT BY RAST MACRO METHOD (STEP BY STEP PROCEDURE):

1. Wash the apparatus
2. Add 5g of the solvent to the specimen tube
3. Heat in water bath and allowed to melt completely.
4. Note the temperature T1 (freezing point).
5. Add 0.5 g of unknown solute to the specimen tube.
6. Stir constantly till it becomes homogenous.
7. Note the freezing point (T2).
8. Difference between the T1 and T2 gives the freezing point of the depression.
9. Knowing the value of Kf molecular weight of the solute is calculated.

EXPERIMENT QUESTIONS

RAST MACRO METHOD：

- What are the applications of the rastmethod .?
- Significance of the rast method when compared to the other methods?
- Why we are specifically adding 5g of the substance to the specimen tube?
- Will the reaction occurs after attaining the homogeneous mixture?
- What is the maximum difference between the t_1 and t_2.?
- When will you note the freezing point of the solution.?

Reference

Video Demonstration

https://www.youtube.com/watch?v=-hFwbpYWWh0

https://www.youtube.com/watch?v=2VzEpsEZOYo(Tamil)

Result

The molecular weight of the given solute= gram.

CALCULATION:

Weight of the weighing bottle with solvent :

Weight of the weighing bottle after transferring the solvent :

Weight of the solvent W_1:

Weight of the weighing bottle with Solute :

Weight of the weighing bottle after transferring the solute :

Weight of the solute W_2:

Freezing point of the pure solvent (T_1):

Freezing point of the solution (T_2):

Freezing point of the depression $\Delta T_f=(T_1-T_2)\ ^{o}C$

Molecular weight of the solute (M_2):

Molal Freezing point constant of the solvent $K_f=\Delta T_f \text{ X } M_2 \text{ X } w_1$

$/(1000 \text{ X } w_2)$

Ex.No:2DETERMINATION OF KF- RAST MACRO METHOD

Aim

To determine the molal depression constant of the unknown solvent using depression in freezing point.

Materials required:

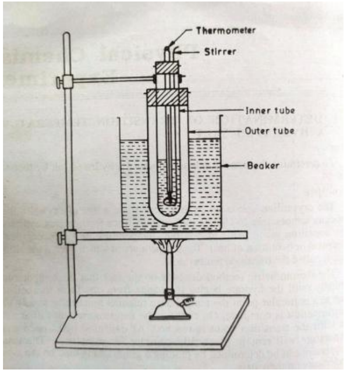
0.5 g of solute; solvent

Principle

When a non-volatile non-electrolyte is dissolved in a non-electrolyte, solvent decrease in freezing point is observed. The depression in freezing point is directly related to the molality of the solution.

$$K_f = \Delta T_f \text{ X } M_2 \text{ X } w_1 /(1000 \text{ X } w_2)$$

Where,



ΔT_f = depression in freezing point

K_f= molal depression constant

M_2 = molecular weight of the solute

w_1 = weight of the solvent

w_2 = weight of the solute

with the value ofdepression in freezing pointK_f of the solvent is determined.

Procedure

Weighed quantity of the given solvent has taken in a specimen tube, surrounded by an air jacket. This was kept in the water bath and allowed to melt completely. The molten liquid was then allowed to cool, with constant stirring the freezing point was noted. Then weighed about 0.5g of the unknown solute accurately and added to the solvent. The mixture was melted and when molten liquid becomes homogeneous it is allowed to freeze with constant stirring and the freezing point of mixture was noted. The depression constant knowing molecular weight the depression constant of the freezing point of given solvent was calculated.

Result

The molal freezing point of the given solvent=……………………….(degree/mole)

Density of Phenol= 1.07

Density of H_2O= 1.00

S.No	Volume Of Phenol(ml)	Volume Of H_2O(ml)	Weight of phenol V X Density 1.01	Weight of H_2O V X Density 1.00	%of phenol	Temperature ^{o}C		
						Heating	Cooling	Average

Ex.No:3DETERMINATION OF CRITICAL SOLUTION TEMPERATURE (PHENOL-WATER SYSTEM)

Aim

To construct the mutual solubility curve for a pair partially miscible liquid namely phenol and water and to determine its critical solution temperature.

Materials required:

Phenol, water, thermometer(110^0 C), round bottom flask

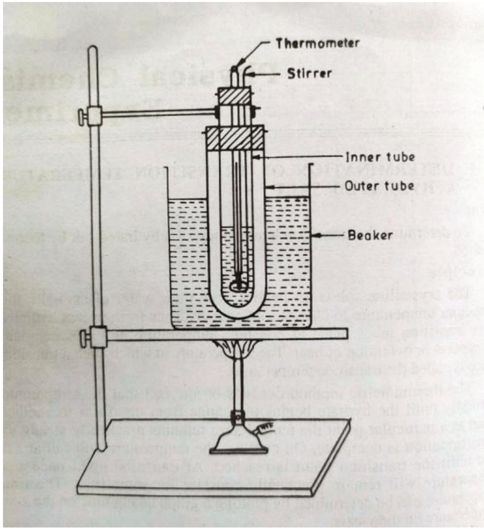
Principle

When two partial miscible liquid pairs such as phenol and water are mixed. Two mutual immisible layers are separated. The upper layer is the solution of water in phenol and the solution of lower layer is phenol in water. At any given temperature, such a system becomes completely miscible into a homogeneous mixture above a certain temperature. This temperature is called the critical solution temperature or consolute temperature.

Procedure

Exactly ………ml of phenol, freshly distilled is taken in a flat bottomed test tube. It is fitted with cork carrying thermometer and stirrer exactly ……… ml of distilled water is added with the help of pipette. The flat bottom tube is placed inside a water bath and slowly heated. The mixture is stirred well and temperature at which the two liquid becomes completed miscible is noted. The mixture is then taken out of the water bath and cooled. The temperature at which the turbidity appears which leads to the separation of two layers is noted. The mean of the two temperature is taken as the miscibility temperature. The experiment is repeated by adding 2,4,6,8,10,12,14,16 ml of water and their corresponding miscibility temperature are noted the miscibility temperature are then plotted against percentage composition of phenol by weight.

The maximum of the curve indicates the critical temperature of the system under consideration.



Experimental Set up

Model Graph

DETERMINATION OF CRITICAL TEMPERATURE – PHENOL WATER SYSTEM (STEP BY STEP PROCEDURE):

1. 5 ml of water is taken in a flat bottom test tube.
2. Set the apparatus as per the figure.
3. With 5ml of water add 1 ml of phenol.
4. Place it in the water bath and heat.
5. Note the heating and cooling temperature.
6. Then add 2,3,4,5 ml of phenol subsequently and repeat the experiment and note the heating and cooling temperature.
7. Discard the solution and wash the apparetus
8. Add 5ml of phenol to the specimen tube
9. Add 3,4,5 ml of water and note the heating and cooling temperature.
10. Plot the graph % of phenol against temperature.

EXPERIMENT QUESTIONS: PHENOL WATER SYSTEM

- What is mean by CST?
- What is the critical solution temperature of phenol water?
- What CST of the system indicate?
- Why phenol and water are immiscible?
- Phenol or water which has more density?
- After attaining CST will the reaction continues?
- What is the IUPAC name of phenol?
- What are the factors that affect CST?
- It is an example for which type of phase system?

REFERENCE

Video Demonstration

https://www.youtube.com/watch?v=5oVnpYhmMVU (Tamil)

$$\% \text{ of phenol} = \frac{Weight\ \ of\ \ \ phenol}{Weight\ \ of\ \ phenol + weight\ of\ water} \text{ X } 100$$

$$= \frac{Volume\ \ of\ \ phenol\ X\ density\ \ of\ \ phenol}{(Volume\ of\ phenol\ X\ Density\ of\ phenol) + (Volume\ of\ H2O + Density\ \ of\ water}$$

Result

The critical solution temperature of phenol water system= …………………………. ^{0}C

The percentage of phenol composition at CST=……………………….%

Calculation

S.No	Volume of phenol (ml)	Volume of NaCl(ml)	Percentage of NaCl	Miscibility Temperature^{0}C		
				Heating	Cooling	Average

Ex.No:4EFFECT OF ELECTROLYTE ON CONSOLUTE TEMPERATURE

Aim

To study the influence of third substance on so consolutetemperature of phenol-water system and to estimate the amount of NaCl in given solution.

Materials required:

Values % of NaCl, phenol, thermometer (110^0 C), round bottom flask

Principle

The consolute temperature of any system is greatly affected by the presence of solute,consolute temperature of the system is raised by addition of sodium chloride.

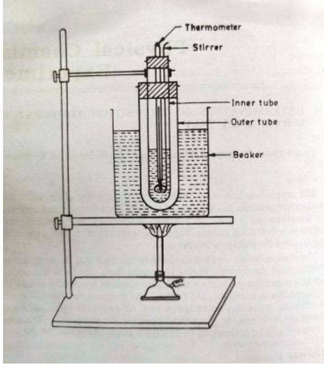
Whenever an electrolyte such as NaCl or KCl is added to a system like phenol-water system the mutual solubility of two layers is affected it has been found that if the electrolyte is capable of dissolving in of the layers, then the CST of phenol-water system is enhanced. This can be verified by taking of solution of NaCl.

Instead of water in the previous experiment and the same procedure is repeated

Procedure

Exactly ……..% of NaCl was prepared and diluted into 0.8%, 0.6%, 0.4%, 0.3%, 0.2% and 1.0 % ………...ml of phenol was taken in a specimen tube and ………ml of ………% NaCl solution was added to it and temperature of complete miscibility was found out. The same procedure was repeated for all other solution of NaCl added at different concentration. The given solution was make up to 100 ml and 5ml of phenol and miscibility point was found out. A graph was drawn by taking % composition on X-axis and temperature along Y-axis. The concentration of unknown sodium chloride was found at extrapolation.

Experimental Setup Model Graph



Result

The concentration of given NaCl solution =**N**

EFFECT OF ELECTROLYTE ON CONSULTE TEMPERATURE (STEP BY STEP PROCEDURE):

1. Prepare 1N NaCl solution.
2. From 1N NaCl solution prepare 0.1, 0.2, 0.3, 0.4, 0.6, 0.8 N solution of NaCl .
3. Set the apparatus as per the figure.
4. Add 5ml of phenol in specimen tube.
5. With that add 5ml of NaCl solution from 0.8, 0.6, 0.4, 0.3, 0.2, 0.1 N solution
6. Note the heating and cooling temperature.
7. Also note the unknown temperature.
8. Plot the graph for concentration of NaCl and Miscibility temperature.

EXPERIMENT QUESTIONS

- What is mean by CST?
- What is the critical solution temperature of phenol water?
- What CST of the system indicate?
- Why phenol and water are immiscible?
- Phenol or water which has more density?
- After attaining CST will the reaction continues?
- What is the IUPAC name of phenol ?
- What are the factors that affect CST?
- It is an example for which type of phase system?

REFERENCE

Video Demonstration

https://www.youtube.com/watch?v=5oVnpYhmMVU (Tamil)

Calculation

% of A=$\frac{Weight of A}{Weight of A+Weight of B}$% of B=$\frac{Weight of B}{Weight of A+Weight of B}$

Calculation

S.NO	Weight of solid(A)(g)	Weight of solid(B(g)	%A	%B	Freezing point ^{o} C

Ex.No:5PHASE DIAGRAM- SIMPLE EUTECTIC SYSTEM

Aim

To construct a phase diagram for a binary mixture and to determine the eutectic temperature and the corresponding composition.

Materials required:

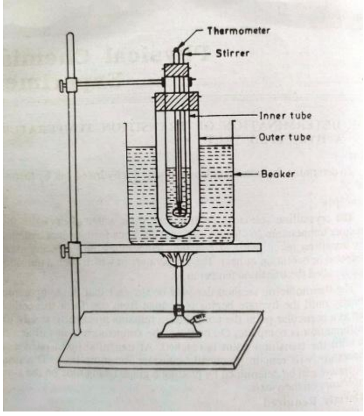
Thermometer; Naphthalene.

Principle

When a binary mixture is cooled solid separates out and the composition of the solid depends upon the nature of the substance. If the components are chemicallydissimilar each lowers the freezing point of the other. The solid crystalline out separately and eutectic system is obtained. The temperature at which the separation results is known as eutectic temperature at which the liquids mixture of the two components will freeze and hence is called eutectic point.

Procedure

A hard glass tube is cleaned and dried, a known accurate weight of solid A (about 5g) is weighed and transferred to it, and sensitive thermometer and a clean stirrer are inserted into the tube. Freezing point of pure solid A is noted. An accurately weighed amount of naphthalene also transferred into it. The heating is continued until the mixture is completely melted and temperature is about 5^0 C higher than the melting point. Now the melt is stirred well to make it attain uniformity of concentration and allowed to cool slowly, stirring should never be stopped at any time. The cooling is continued until a solid phase begins to crystallize out. The temperature of the appearance of the first crystal is noted. The experiment is repeated in various properties varying from 10% to 90% of each of the components are so taken that of total weight of the mixture remains constant (5g). The freezing point of the pure solid B is found out. The freezing point is plotted against their corresponding composition. A graph is drawn with % composition of x-axis and freezing point of Y-axis. The resulting graph has minimum temperature point, which is the eutectic point. The point of actually got by drawing smooth, free hand occurs through the plotted points on either side of the eutectic and freezing point where they meet. The eutectic composition is read from the graph.



Result

The eutectic temperature and composition of solid A, solid B was found to be

i) Eutectic temperature = ……………………………….^{O}C

ii) Eutectic composition =………………………………..%

PHASE DIAGRAM – SIMPLE EUTECTIC SYSTEM (STEP BY STEP PROCEDURE):

1. Take almost or exactly 5g of solid A in a specimen tube. Solid A constant
2. Heat in water bath.
3. Note freezing point.
4. Then add 1,2,3,4,5g of Solid B to Solid A.
5. Stir well to get homogenous solution.
6. Note the freezing point.
7. Discard and again wash the apparatus thoroughly.
8. Take 5g of solid B in a specimen tube. Solid B is constant.
9. Add 1,2,3,4,5g of Solid A to specimen tube.
10. Stir well to get homogenous solution.
11. Note the freezing point
12. Using formula the % of A and % of B is found.
13. Plot the graph against composition and freezing point.

EXPERIMENT QUESTIONS

- What is phase diagram .
- What is Binary mixture.
- Explain eutectic temperature.
- Lowers the freezing point - Explain.
- Explain – Eutectic Point, Eutectic System.

Oh heating

On cooling

S. No	Time(Sec)	Temperature^{0}C	S. No	Time(Sec)	Temperature^{o}C

Ex.No:6 DETERMINATION OF TRANSITION TEMPERATURE OF HYDRATED SALT

Aim

To determine the transition temperature of hydrated salt by thermometric method.

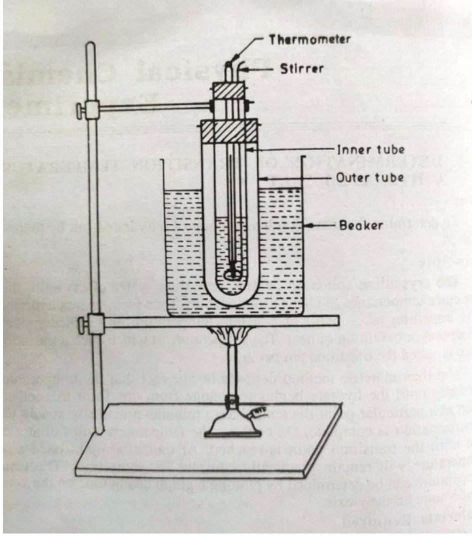
Materials required:

Stopwatch; Thermometer

Principle

The crystalline solids on heating lose their water of crystallization at a particular temperature to change into anhydrous form. The transition takes place at constant temperature and it accompanied by evolution of heat. The temperature at which such transition takes place is called transition temperature.

The thermometric methods depend on the fact that temperature is normally until the hydrate begins to change from one form to another form. Then at a particular point, the temperature remains practically steady until the transformation is complete. On cooling,the temperature will fall at definite rate until the transition point is reached. At transition point, once again the temperature will remain practically constant for some time. The transition temperature can be determined by plotting a graph taking time on the x-axis and temperature on the y-axis.



Materials required:

- A hydrated salt
- Transition temperature apparatus
- A sensitive thermometer
- Stirrer
- Water bath
- Stopwatch

Procedure

The given hydrated salt along with a few ml of a non-polar solvent such as paraffin Liquid is placed in the inner tube of the transition temperature apparatus, fitted with a stirrer and thermometer. The bulb of thermometer should be kept immersed in the sample. The inner tubes then placed inside the outer jacket and this arrangement is immersed in water bath. The temperature of the water bath was slowly raised by using same flame. The rate of increase in temperature of the salt is kept uniform by slow and regular stirring. The kept temperature is recorded for every half a minute. The temperature increases gradually remains constant for some time and again increases.The transition temperature apparatus is taken out of the water bath. It is allowed to cool slowly with constant stirring. The temperature is recorded for every half of minute. At one point, the temperature remains constant and the cooling is continued for some more time. A graph is plotted by taking time in the abscissa and temperature in the ordinate. 2 curves will be obtained one for heating and other for cooling. The curve has an approximately horizontal position. The temperature corresponding to the horizontal position in graph is noted. This give the transition temperature of a given salt hydrate.

Result

The transition temperature of cooling =^{0}C
The given salt hydrate on heating = ^{0}C

S.No	Time (min)	Volume of NaOH Solution (ml)	$(V\infty\text{-}Vt)$ (ml)	$\text{Log}(V\infty\text{-}Vt)$	$K=\frac{2.303}{t}\log\frac{(V\infty - Vo)}{(V\infty - Vt)}*$ 10^{-3} (min^{-1})

Calculation

1) $K=\frac{2.303}{t}\log\frac{(V\infty - Vo)}{(V\infty - Vt)}\text{min}^{-1}$

V_{∞} = volume of sodium hydroxide at infinite time (ml)

Alternatively a graph is plotted well log (V_{∞} - V_t) against time in minutes. A straight line is drawn through the point. Its slope is determined an value of k is calculated from the slope using the expression.

Ex.No:7 ACID CATALYZED HYDROLYSIS OF AN ESTER

Aim

To determine the rate constant for the acid catalyzed hydrolysis of an ester (methyl acetate or ethyl acetate) at room temperature using hydrochloric acid as a catalyst.

Materialsrequired:

HCl = 0.5 N; NaOH = 0.1 N; Ester = 10ml; phenolphthalein

Principle

When an ester such as methyl acetate or ethyl acetate reacts with water it is converted into an acid and alcohol. The hydrolysis takes place slowly and it is catalyzed by an acid and the velocity depends upon the strength of an acid. The reaction is pseudounimolecular, when water present in excess. rate = $- dc/dt = k'_1[\text{Ester}]$

Where,C= concentration of the ester at any time ‘t’K_1= rate constant

The accumulation of acetic acid increases as the reaction progresses. It can be followed by drawing a known volume and the reaction at known regular intervals and titrating it against standard NaOH solution.

Procedure

100ml of N/2 Hydrochloric acid is taken in dry conical flask and kept into a water bath at a room temperature. The ester is kept at the same temperature in a boiling test tube. After half an hour 10ml of ester is pipetted out into a conical flask containing acid zero time is noted when half the volume of ester solution is transferred into the reaction bottle. The contents are shaken well and immediately 5ml of this reaction mixture is withdrawn by means of dry pipette and poured into a clean conical flask containing ice pieces and a drop of phenolphthalein. The acid liberated is titrated against approximately N/5 sodium hydroxide solution the titre value gives V_o similarly the titration are repeated at regular intervals of 10 minutes upto 60 minutes the final titration value can be done by heating remaining solution in water heater at about 60 to 70^0 C for 90 minutes to the reaction to complete.

This is than cooled at room temperature and the titration is repeated as before the velocity constant is calculated using the following formula

$$K = 2.303/t \log (V_\infty - V_0)/(V_\infty - V_t) \text{ min}^{-1}$$

Where, t = time in minutesV_t = volume of sodium hydroxide at corresponding time (ml)
V_0 = volume of sodium hydroxide at zero time (ml)

Result

The rate constant of the acid catalyst hydrolysis of an ester

Kcal= …………………………..$X10^{-3}$ min^{-1}

K graph= …………………………..$X10^{-3}$ min^{-1}

ACID CATALYSED HYDROLYSIS OF AN ESTER (STEP BY STEP PROCEDURE):

1. Take 100ml – 0.5N HCl in a reaction bottle.

2. Kept into a water bath (dust container) with tap water.
3. Take exactly 10ml of ester in a boiling tube.
4. Place boiling tube and the reaction bottle in the water bath.
5. Keep the solution for 20 mins in a water bath.
6. Between the time gap get prepare for the titration(Fill NaOH in burrete)
7. In conical flask add 2 cubes of ice and add a drop of phenolphthalein indicator.
8. Transfer ester 10ml from the boiling tube to the reaction bottle, shake gently, for even mixing. Switch on the stop watch for time in mins.
9. Immediately take 5ml of reaction mixture and transfer into conical flask.
10. Start the titration immediately.
11. The end point is appearance of permanent pale pink colour.
12. After each 10mins time interval repeat the titration by taking the solution from the reaction bottle and titrating against the NaOH solution .
13. After first titration take around 40ml reaction mixture in a conical flask and place it hot water bath (60-70 degree C). This is for the infinity titration.
14. Repeat the titration for 60 mins (0, 10, 20, 30, 40, 50, 60)
15. After 1hour take 5ml from step 13 solution.
16. Again titrate for infinity reading.
17. Plot the graph for time against log (V_∞ - V_t).

HYDROLYSIS OF AN ESTER

- What is mean by acid hydrolysis ?
- What are the products of the acid hydrolysis of an ester?
- What happens when ester reacts with hcl?
- Why ester gets evaporated?
- What is the mechanism of an ester hydrolysis?
- Why we are adding ice cubes and phneolpthalein to the reactant ?
- How will you find the infinity value?
- Why we are keeping the ester in boiling water bath ?
- At what time we have to start the stop clock?

S.NO	Concentration Of $KMnO_4$(N)	Absorbance (A)

Calculation

$V_1N_1=V_2N_2$

Ex.No:8 ESTIMATION OF MANGENESE BYCOLOROIMETRY METHOD

Aim

To test the validity of beer's lambert's law using a photo-electriccolorimetry and to determine the unknown concentration.

Materials required:

0.1N KMnO4 in 250 ml; Eq. Wt. of KMnO4 is 31.6; Molecular weight is 158.0

Principle

According to beer lambert's law

$A = \varepsilon cl$

A is the absorbance or optical density

$\varepsilon$is the molar absorption co-efficient

C is the concentration of absorbing solution in $moldm^{-3}$

l is the length of the cell

A plot of A versus C must be linear if that beer- lambert's law is valid from the calibration curve. So obtained the concentration of unknown solution can be determined by measuring its absorbance.

Procedure

A stock solution of potassium permanganate was prepared and is diluted quantitatively in the range of $0.7X\ 10^{-7}$ M to $2.5\ X\ 10^{-4}$M. By using the appropriate filter 530 nm the absorbance of above solutions were measured. Absorbance was plotted against concentration. The absorbance of given unknown solutions were measured corresponding to this optical densities. The concentration of unknown can be determined from the calibration curve.

Result

i) The linear relationship between absorbance and concentration indicates the validity of beer-Lambert's law

ii) The concentration of unknown $KMnO_4$ is =

CALORIMETRY(STEP BY STEP PROCEDURE:

1. Prepare 0.0001, 0.0002, 0.0003, 0.0004, 0.0005, 0.0006 N solution of $KMnO_4$ from the stock solution.
2. Set the spectrometer at 620nm and nullify by adding water.
3. Add the 0.0001, 0.0002, 0.0003, 0.0004, 0.0005, 0.0006 N $KMnO_4$ to the sample tube and note the absorbance
4. Plot graph against concentration and absorbance.

EXPERIMENTAL QUESTION

Reference

Weight of weighing bottle before transfer =

Weight of weighing bottle after transfer =

Weight of Ferrous ammonium Sulphate =

Normality of FAS =Weight of FAS/gram equivalent *Volume in lit

S. No	Volume of $KMnO_4$(ml)	EMF(mv)

S. No	Volume of $KMnO_4$(ml)	EMF(mv)

Ex.No:9 POTENTIOMETRIC TITRATION REDOX TITRATION [$KMnO_4$ Vs FAS]

Aim

To determine the strength of $KMnO_4$ using standard ferrous ammonium sulphate by potentiometric titration.

Materials required:

FAS – Eq. Wt. -392.2 g; 1N FAS in 1 Litre – 392.2 g; 0.25 N FAS in 250 ml – 24.51 g; 1N FAS in 250 ml – 98.05 g; KMnO4 – 1N KMnO4 in 1 Litre = 31.6 g; 0.5 N in 2 litres = 31.6 g; FAS = N/40 or N/20 Vs KMnO4 = N/20 or N/40

Principle

Potassium permanganate oxidies the ferrous ion into ferric ions in the presence of acid. The reaction involved is

$$KMnO_4^- + 8H^+ + 5e^- \longrightarrow Mn^{2+} + 4H_2O$$

$$5Fe^{3+} \longrightarrow 5Fe^{3+} + 5O^-$$

The electrode potential is this titration depends upon the concertation of Fe^{2+}, Fe^{3+} and H^+ ions to avoid the effect of change in H^+ ions. Concentration of electrode potential the titration as usually carried out in a presence of large access in acid the oxidation potential of redox reaction

$$E= E^0 – [RT/F] \ln [Fe^{3+}]/[Fe^{2+}]$$

The necessary cell is setup connecting the redox against a calomel electrode as shown in saturated calomel electrode, salt Fe^{2+} , Fe^{3+} and H^+. When $KMnO_4$ is added to the system Fe^{2+} is converted to Fe^{3+} whose concentration increases with progressive adding $KMnO_4$ the order observed gradually increase at the end point there will be a change due to sudden removal of Fe^{2+} ions. Plot between emf measured E against the volume of $KMnO_4$ added is shown and the endpoint is determined graphically.

Procedure

Accurately 0.1 N ferrous ammonium sulphate solution was prepared and 10ml of this was pipetted out into a beaker equal amount of dilute sulphuric acid was added.

A platinum electrode was dipped into the solution and connected to reference electrode.Calomel electrode (reference electrode) dipped in KCl solution in another beaker which is connected through salt bridge.

For each addition of potassium permanganate solution, the corresponding emf value was noted from the value of equivalent point 0.5ml, 0.2 ml and 0.1 ml portion of titrate were added.

The following graph were drawn

1. EMF Vs Volume of $KMnO_4$

2. ΔE Vs Mean Volume of $KMnO_4$

POTENTIOMETRIC TITRATION- ACID BASE TITRATION (STEP BY STEP PROCEDURE):

1. Wash the apparatus.
2. Prepare saturated KCl solution- insert calomel electrode (reference electrode)
3. 0.1 N of HCl is prepared.
4. 20 ml is pippeted out in a beaker- add a pinch of Quinhydrone- insert standard electrode (platinum electrode).
5. Salt bridge is connected between the beaker (100 ml)
6. 0.1N of NaOH prepared- bureete solution.
7. NaOH is proportionally added to HCl.
8. Note the reading and plot graph (S shape).
9. Value gradually decreases and becomes negative.
10. Then NaOH is added 0.1 ml, 0.2 ml….
11. Plot the graph and Note the peak value.
12. Using the peak value strength of NaOH is calculated.

EXPERIMENTAL QUESTION POTENTIOMETRIC TITRATIONS

- What is an potentiometer?
- What is mean by emf?
- Why salt bridge where used in the potentiometer?
- What is mean by salt bridge?
- What are the applications of potentiometry?
- Why the value decrease after attaining the equivalent point in acid base titrations?
- What are the electrodes used in the potentiometric titrations?
- What is reference electrode? What is calomel electrode?
- Why KCl solution were used in acid base titration?
- Why we are adding quinhydrone in acid base titration?
- What is an acid base titration?
- What is mean by redox titration?
- Why we are keeping read mode in the potentiometer?
- Why the EMF suddenly increases after attaining the equivalent point .?
- How will you determine the equivalent point of the reaction?
- What are the significance of potentiometric titrations?

EXPERIMENTAL SETUP

MODEL Graph

Reference

S. No	Volume of $KMnO_4$(ml)	EMF(mv)

S. No	Volume of $KMnO_4$(ml)	EMF(mv)

S.NO	**Volume of $KmNO_4$(ml)**	**EMF(mv)**	**ΔE**	**ΔV**	$\frac{\Delta E}{\Delta V}$**=mv/ml**	**Mean value of $KmNO_4$ (ml)**

S.NO	**Volume of $KmNO_4$(ml)**	**EMF(mv)**	**ΔE(ml)**	**ΔV(ml)**	**$\frac{\Delta E}{\Delta V}$=mv/ml**	**Mean value of $KmNO_4$ (ml)**

Volume of FAS Solution V_1=

Normality of FAS Solution N_1=

Volume of $KmNO_4$ V_2=

Normality of $KmNO_4$ $N_2=V_1N_1/V_2$

Result

The strength of potassium permanganate solution =

Volume of NaOH (V1) =

Strength of NaOH(N1) =

Volume of HCl (V2) =

Strength of HCl (N2) = V1XN1/V2

S. No	Volume of NaOH added (ml)	Observed conductance Ohm^{-1}

Ex.NO:10CONDUCTOMETRIC TITRATION-
STRONG ACID Vs STRONG BASE TITRATION(HCl vs NaOH)

Aim

To determine the strength of given hydrochloric acid solution by titration with standard NaOH by conductometric titration.

Materials required:

Stock Solution :NaOH = 0.5 N; HCl = 0.2 N

Principle

Solution of electrolysis conduct electricity due to the presence of ions. The specific conduction of solution is proportional to the concentration of ions in it. Reaction between HCl and NaOH, may be represented as,

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

When a solution of HCl acid is treated with NaOH. The fast moving hydrogen ions are progressively replaced by slow moving Na^+ ions as a result. Conductance will takes place until the end point is replaced further, sharply there is an excess of free OH^- ions. A graph is drawn between volume of NaOH added and the conductance of solution. The exact end point is the point of inert section at the two curves.

Procedure

The burette is filled withstandard NaOH solution, exactly 20ml of the given HCl acid solution is pipetted out into a clean beaker. A glass rod is dipped in the beaker. The conductivity cell is dipped in 0.5 ml portion from the burette.

After each addition there is attainment of process is repeated until at least the reading taken behind the endpoint. The values of observed conductance are plotted against volume of NaOH added. The end point of could be obtained from the point of intersection.

CONDUCTOMETRIC TITRATION (STEP BY STEP PROCEDURE):

1. Apparatus should be cleanly washed.
2. Nullified to zero.
3. 0.1N NaOH is prepared- burrete solution.
4. 0.1N HCl solution is prepared- pippete solution.
5. 20ml of HCl is taken in beaker.
6. Conductivity cell is dipped in the beaker with the glass rod to stir.
7. NaOH is proportionally added to the beaker and readings are noted.
8. Initially the value decreases.
9. At certain point suddenly the conductance increases.
10. Plot the graph.
11. Point of intersection of graph is noted.
12. Using that point strength of HCl is calculated.

CONDUCTOMETRIC TITRATIONS

- What is the unit of conductance ?
- What is the equivalent weight of HCl and NaOH?
- How the conductance is related to the concentrations of the ions?
- Why conductance decrease on addition of NaOH to HCl?
- Define conductance.
- What is the difference between conductometric titration and potentiometric titrations?
- When strong acid combines with strong acid what type of reaction occurs?
- Which electrode were used in the conductometric titrations?
- What could be the shape of the graph?
- What is the principle of conductometric titrations?

EXPERIMENTAL SETUP

MODEL Graph

Reference https://www.youtube.com/watch?v=-GS6uoFf3qQ

S. No	Volume of NaOH added (ml)	Observed conductance Ohm^{-1}

S. No	Volume of NaOH added (ml)	Observed conductance Ohm^{-1}

Result

The strength of given HCl=

Determination of cell constant

S.No	Concentration of KCl	Conductance (Ω-1)or ohm-^{1}	Specfic Conductance ohm^{-1}cm-^{1}	Cell Constant =specific conductance /observed Conductance cm-^{1}

λ_{0} = 390.20Ohm^{-1}cm^{2}gequ^{-1}

Determination of Dissociation constant

S.No	Concentration of Acetic acid (C)	Conductance *10^{-4} ohm^{-1}	Specific conductanceK=$\frac{1}{R}$*Cell constant ohm^{-1}cm-^{1}	$\Lambda c=\frac{1000*K}{C}$ ohm-^{1}cm–2eq^{-1}	α$\frac{\Lambda c}{\Lambda 0}$	Kα=$\frac{C\alpha 2}{1-\alpha}\times 10^{-5}$

Average

Ex.No:11 DISSOCIATION CONSTANT OF A WEAK ACID

Aim

To determine the equivalent conductance of the weak electrolyte at various concentration and its dissociation constant.

Materials required: Acetic acid – 1N; Lab Normality – 17.4 N ; 1N in 250 ml = (1N * 250 ml) / 17.4;
1N Acetic Acid in 250ml = 14.36 ml; From 17.4 N Further Dilution to N/10 = 0.1 N

Principle

The equivalent is product of absorbed specific conductance and the value inml containing 1g equivalent of electrolyte. The equivalent conductance of strong electrolyte do not vary much with concentration. In the case of weak electrolyte, equivalent conductance increases and specific conductance decreases with dilution. The equivalent conductance of a weak electrolyte(Λ_C) at different concentration is determined by measuring the resistance of this solution plot of (Λ_C) vs $\sqrt{2}$ result in the parabola.

The degree of dissociation(α) of the electrolyte at the different concentration can be obtained by the equation $\alpha = \frac{\lambda C}{\lambda \infty}$

Λ_0 = equivalent conductance of the weak electrolyte at infinite solution

The dissociation constant of the weak electrolyte is given by ostwald's dilution law

$$K_C = c\alpha^2 / (1-\alpha)$$

By knowing, the value of C and α, K_a is determined.

Procedure,

Exactly N/10 KCl solution is prepared and its resistance is measured. From its value the cell constant of the given conductivity cell is calculated.

A 0.1N solution of a weak electrolyte say acetic acid is prepared using conductivity water (its exact strength is determined by titrating against standard NaOH solution using Phenolphthalein indicator). From this stock solution 0.02, 0.04, 0.05, 0.06, 0.08N are prepared by using conductivity water each solution is then taken in a beaker and then conductivity cell whose cell constant is determined previously is dipped into it. The reversibility of the solution is measured by Conductivity Bridge.

From the measured resistance and cell constant, the specific conductance of each solution is calculatedand hence conductance is calculated. Using the equivalent conductance at infinite dilution Λ_0 of acetic acid is the degree of the dissociation (α) is calculated using Ostwald dilution law .

DISSOCIATION CONSTANT OF A WEAK ACID (STEP BY STEP PROCEDURE):

1. Wash the apparatus in deionized water.
2. Prepare 0.1N KCl in deionized water.
3. Prepare 0.1N $CH3COOH$ in deionized water.
4. From 0.1N $CH3COOH$, 0.01, 0.02, 0.04, 0.06 and 0.08N $CH3COOH$ to be prepared.
5. Conductance of KCl is noted from conductometric instrument for cc measurement.
6. Value of specific conductance is 0.01412.
7. Calculate conductance of $CH3COOH$ for various concentration
8. Value of R=
9. Value of **λ_0**= 390.202 Ω^{-1} cm^2 g equ^{-1}
10. From the known values K∞ is calculated.
11. Plot the graph

INSTRUMENT SETUP:

- Conductivity cell and note the cc (cell constant)
- Connect the cell properly, and check the reading weather it is change while taking it away from the solution.
- Select Read mode (conductance).
- Sensitivity option select.

DISSOCIATION CONSTANT OF A WEAK ACID

- What is mean by conductance?
- What is the difference between specific and observed conductance?
- What is the value of specific conductance of acetic acid and how will you calculate it?
- What is mean by cell constant?
- What are the applications of this experiment?
- How will you determine K∞?
- Why we are using 0.01 etc normality solutions in this experiment? (is this experiment depends upon the concentration?
- How will you find the $\Lambda 0$?

Calculation

$V_1N_1= V_2N_2$

Result

The dissociation constant of the given

weak electrolyte (CH_3COOH) = ………………………………...OHM^{-1}cm^{2}gequ^{-1}

Conc.Of KCl(N)	Known Resistance R (Ω)	Ax (cm)	Bx(cm)	Rx=$\frac{Ax}{Bx}$*R(Ω)	Cell Constant= specific conductance/observed conductance cm–1

Observed conductance = 1/ Resistance

Cell Constant = Specific Conductance / Observed Conductance

Ex.No:12DETERMINATION OF EQUIVALENT CONDUCTANCE OF STRONG ELECTROLYTE

Aim

To determine the equivalent conductance of strong electrolyte solution of various concentration and infinite solution

Materials required:

Stock Solution 1N Acetic acid: Experiment solution 0.01N, 0.02N, 0.03N, 0.04N.

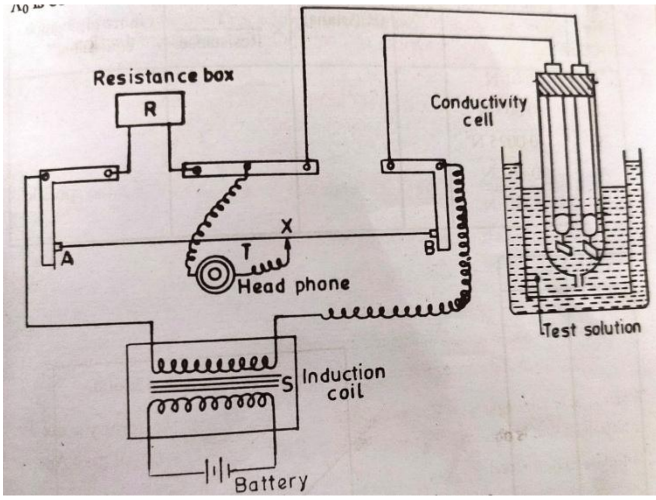
Principle

The equivalent conductance of an electrolyte is given by

$\Lambda = 1000 X k /C$

Where

C = concentration of given equivalent per time
k = specific conductance
λ = equivalent conductance
According to debye-Huckelonsagar equation
$\lambda = \lambda_0 - (A-B) \sqrt{2}$
Where, A,B –conmstan for given electrolyte
λ_0-concentration of electrolyte at infinite dilution



Procedure

Exactly 0.1 N solution of the given strong electrolyteKCl or NaOH is prepared by different volume of solution. It is diluted to obtain a range of concentration of solution of electrolyte such as 0.01, 0.02, 0.03 & 0.04N.

The resistance of solution is determined by taking the solution in a conductivity cell of known cell constant and connecting it to a conductivity bridge. From the value of resistance measured of a particular concentration solution is equivalent conductance is calculated. A graph is drawn between Λ and $\sqrt{C}$, by extrapolation to $\sqrt{C} = 0$, $\lambda_0$is obtained.

EQUIVALENT CONDUCATNCE OF STRONG ELECTROLYTE:

- Why we are using KCl as an electrolyte?
- How will you prepare 0.1 n KCl?
- What is an electrode?
- What is the role of an electrode in tjis experiment?
- Why the sound increases when the concentration of the solution increases ?
- What is the major application of this experiment?
- How will you calculate the Bx value?

Titration 1

S.No	Concentration of KCl(ml)	Known resistance R(Ω)	Ax(cm)	Bx(cm)	Rx=$\frac{Ax}{Bx}$*R(Ω)

Titration 2

S.No	Concentration aq.KCl (C)	$\sqrt{c}$	Resistance (R)Ω RX	K=Cell constant *$\frac{1}{Rx}\Omega/cm$	$\Lambda=\frac{1000*K}{C}$ Ω– 1cm^{2}eqv^{-1}

Calculation:**V_1N_1=V2N_2**

Result:

The equivalent conductance of given electrolyte at various concentration

i) λ(0.01)=
ii) λ (0.02)=
iii) λ (0.03)=
iv) λ (0.04)=
v) λ (0.05)=
vi) λ (0.06)=

The equivalent conductance of strong electrolyte at infinite dilution λ_0 =

S. No	Volume of NaOH(ml)	Emf(mv)

S. No	Volume of NaOH(ml)	Emf(mv)

Ex.No:13POTENTIOMETRIC TITRATION ACID-BASE TITRATION

Aim

To determine the strength of sodium hydroxide using standard HCl by potentiometric titration

Materials Required:

HCl Strong acid – Lab Normality = 11.3 N; NaOH Unknown Approximately 0.1N; Stock solution HCl = 0.2 N; NaOH = 0.5 N

Principle

Solution of electrolyte conducts electricity due to the presence of ions. The Specific conductance at a solution is proportional to the concentration of the ions in it. The reaction between hydrochloric acid and sodium hydroxide may be represented as sodium hydroxide.

When a solution of hydrochloric acid is treated with sodium hydroxide. Fast movingH^+ions progressively replace the slow moving Na^+ ions. As a result, conductance of the solution decrease will take place until the end point is reached. Further addition of alkali raises the conductance sharply as there is an excess of hydrogen ions. Graph is shown between volume of sodium hydroxide and the conductance of solution. The exact point is the point of intersection of two curves.

Procedure

Accurately 0.1 N hydrochloric acid was prepared. 20 ml of this acid was pipetted out into a beaker A Point of quinhydrone was added. A platinum electrode was dipped into a solution and was connected to a reference electrode (calomel electrode) in another beaker through a salt bridge. The given sodium hydroxide was taken in a burette. After each addition EMF is noted. An accurate experiment was carried out by noting the EMF and on heating the equalivalent point 0.5 ml,0.2ml and 0.1ml portion of the titration.

The following graph were drawn

i) EMF vs volume of sodium hydroxide

ii) $\Delta E/\Delta V$ Vs mean volume of sodium hydroxide

POTENTIOMETRIC TITRATION- ACID BASE TITRATION (STEP BY STEP PROCEDURE):

1. Wash the apparatus.
2. Prepare saturated KCl solution- insert calomel electrode (reference electrode)
3. 0.1 N of HCl is prepared.
4. 20 ml is pippeted out in a beaker- add a pinch of Quinhydrone- insert standard electrode (platinum electrode).
5. Salt bridge is connected between the beaker (100 ml)
6. 0.1N of NaOH prepared- bureete solution.
7. NaOH is proportionally added to HCl.
8. Note the reading and plot graph (S shape).
9. Value gradually decreases and becomes negative.
10. Then NaOH is added 0.1 ml, 0.2 ml….
11. Plot the graph.
12. Mote the peak value.
13. Using the peak value strength of NaOH is calculated.

POTENTIOMETRIC TITRATIONS

- What is a potentiometer?
- What is mean by emf?
- Why salt bridge where used in the potentiometer?
- What is mean by salt bridge?
- What are the applications of potentiometry?
- Why the value decrease after attaining the equivalent point in acid base titrations?
- What are the electrodes used in the potentiometric titrations?
- What is reference electrode?
- What is calomel electrode?
- Why KCl solution were used in acid base titration?
- Why we are adding quinhydrone in acid base titration?
- What is an acid base titration?
- What is mean by redox titration?
- Why we are keeping read mode in the potentiometer?
- Why the value suddenly increases after attaining the equivalent point?
- How will you determine the equivalent point of the reaction?
- What are the significance of potentiometric titrations?

Calculation

Volume of NaOH $V_2=$

Volume of HCl $V_1=$

Strength OfHCl $N_1=$

Strength Of NaOH $N_2= V_1 N_1/ V_2$

S.NO	**Volume of NaOH(ml)**	**EMF(mv)**	**∆E(mv)**	**∆V(ml)**	**$\frac{\Delta E}{\Delta V}$=mv/ml**	**Mean value of NaOH(ml)**

S.NO	Volume of NaOH(ml)	EMF(mv)	∆E(mv)	∆V(ml)	$\frac{\Delta E}{\Delta V}$=mv/ml	Mean value of NaOH(ml)

Result

The strength of the given sodium hydroxide solution N_2 =________N

S.NO	**Concentration Of $CuSO_4$(N)**	**Absorbance (A)**

Calculation

$V_1N_1=V_2N_2$

Ex.No:14ESTIMATION OF COPPER BY COLORIMETRY METHOD

Aim

To test the validity of beer's lambert's law using a photo-electriccolorimetry and to determine the unknown concentration.

Materials required:

Stock solution of potassium permanganate; Colorimetry

Principle

According to beer lambert's law

$A= \varepsilon cl$

A is the absorbance or optical density

$\varepsilon$is the molar absorption co-efficient

C is the concentration of absorbing solution in $moldm^{-3}$

l is the length of the cell

A plot of A versus C must be linear if that beer- lambert's law is valid from the calibration curve. So obtained the concentration of unknown solution can be determined by measuring its absorbance.

Procedure

A stock solution of potassium permanganate was prepared and is diluted quantitatively in the range of $0.7X\ 10^{-7}$ M to $2.5\ X\ 10^{-4}$M. By using the appropriate filter 530 nm the absorbance of above solutions were measured. Absorbance was plotted against concentration. The absorbance of given unknown solutions were measured corresponding to this optical densities. The concentration of unknown can be determined from the calibration curve.

Result

i) The linear relationship between absorbance and concentration indicates the validity of beer-Lambert's law

ii) The concentration of unknown $CuSO_4$ is =

S.NO	**Concentration Of $FeSO_4$(N)**	**Absorbance (A)**

Calculation

$V_1N_1=V_2N_2$

Ex.No:15ESTIMATION OF IRON BY COLORIMETRY METHOD

Aim

To test the validity of beer's lambert's law using a photo-electriccolorimetry and to determine the unknown concentration.

Materials required:

Stock solution of Potassium Permanganate ;Colorimetry;

Principle

According to beer lambert's law

$A = \varepsilon cl$

A is the absorbance or optical density

$\varepsilon$is the molar absorption co-efficient

C is the concentration of absorbing solution in moldm^{-3}

l is the length of the cell

A plot of A versus C must be linear if that beer- lambert's law is valid from the calibration curve. So obtained the concentration of unknown solution can be determined by measuring its absorbance.

Procedure

A stock solution of potassium permanganate was prepared and is diluted quantitatively in the range of 0.7X 10^{-7} M to 2.5 X 10^{-4}M. By using the appropriate filter 530 nm the absorbance of above solutions were measured. Absorbance was plotted against concentration. The absorbance of given unknown solutions were measured corresponding to this optical densities. The concentration of unknown can be determined from the calibration curve.

Result

i) The linear relationship between absorbance and concentration indicates the validity of beer-Lambert's law

ii) The concentration of unknown $FeSO_4$ is =