V.V.VANNIAPERUMAL COLLEGE FOR WOMEN
VIRUDHUNAGAR

LAB MANUAL FOR PHYSICAL CHEMISTRY EXPERIMENTS

(UNDER DBT STAR COLLEGE SCHEME)

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V.V.Vanniaperumal College for Women, Virudhunagar Tamilnadu.
No HRD-11011/163/2020-HRD-DBT-Chemistry/Lab Manual 1

V.V.VANNIAPERUMAL COLLEGE FOR WOMEN

(Belonging to Virudhunagar Hindu Nadars)
An Autonomous Institution Affiliated to Madurai Kamaraj University
Re-accredited with 'A' Grade (3rd cycle) by NAAC
VIRUDHUNAGAR – 626 001 (TAMIL NADU)

DBT STAR COLLEGE SCHEME

Department of Biotechnology, Ministry of Science and Technology
Government of India, New Delhi

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FOREWORD

The Lab Manual for **"PHYSICAL CHEMISTRY EXPERIMENTS"** is designed to acquaint the students with essential skills and techniques in accordance with the updated syllabus under DBT Star College Scheme sponsored by the Department of Biotechnology, Ministry of Science and Technology, MHRD, New Delhi. The skill of handling instruments and performing experiments will reinforce the theoretical knowledge of learnt concepts.

We thank the **Department of Biotechnology, The Ministry of Science and Technology, MHRD, New Delhi** for providing a good opportunity under Star College Scheme (No HRD11011/163/2020-HRD-DBT Dt. 24.08.2020). Under this scheme, we have purchased pH meter, UV photoreactor, Analytical balance, Colorimeter, Distillation Unit, Chemicals and Glassware. This provision enables the students for better understanding of basic concepts in Chemistry and to develop curiosity for further progress.

We hope this manual will surely fulfilthe student's need to enhance their attitude towards research and empower them as a better chemist.

MEMBER SECRETARY / COORDINATOR **CHAIRMAN / PRINCIPAL**

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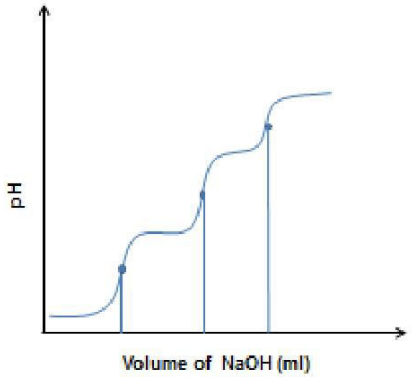
1. DETERMINATION OF I,II, III ORDER DISSOCIATION CONSTANT OF H_3PO_4 BY pH MEASUREMENT

S.No	Volume of NaOH (ml)	pH



In order to determine the III order dissociation constant of phosphoric acid, 5ml of 0.3M $CaCl_2$ solution was added and the titration was continued until there is sharp rise in the pH value was observed.

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1. DETERMINATION OF I,II, III ORDER DISSOCIATION CONSTANT OF H_3PO_4 BY pH MEASUREMENT

AIM:

To determine the I,II, III order dissociation constant (pKa) of H_3PO_4 by pH measurement.

REQUIREMENTS:

EQUIPMENTS	: pH Meter, Glass Electrode
CHEMICALS	: Buffer tabletsfor pH meter calibration, ortho phosphoric acid, sodium hydroxide, phenolphthalein
GLASSWARES	: Volumetric flask (100 ml), burette, pipette, weighing bottle

PRINCIPLE:

The quantitative measure of the strength of an acid is measured from its pH value. The strength of an acid (HA) is defined by its ability to donate a proton and form conjugate base A^-. Higher the number of hydrogen ions liberated per mole of acid in solution, higher the strength of the acid.

$$HA + H_2O \rightleftharpoons H_3O^+ + A^-$$

According to Henderson – Hasselbach equation, the relationship between pH and pKa value for a weak acid, HA.

$$pH = pK_a + \log\frac{[A^-]}{[HA]}$$

when the concentration of the acid and the anion becomes equal pH = pKa; in other words, at the pH equal to the pKa the concentrations of the acid and the conjugate base form are equal. This is the principle to determine the pKa.

At the point of half neutralization, $[A^-]=[HA]$. Therefore

$pK_a=pH$

Phosphoric acid is a tribasic acid and neutralised in three stages as follows.

$$H_3PO_4 + H_2O \rightleftharpoons H_3O^+ + H_2PO_4^-$$
$$H_2PO_4^- + H_2O \rightleftharpoons H_3O^+ + HPO_4^{2-}$$
$$HPO_4^{2-} + H_2O \rightleftharpoons H_3O^+ + PO_4^{3-}$$

The successive ionization constants, neglecting the activity coefficient terms are,

$$pKa_1 = \frac{[H_2PO_4^-][H_3O^+]}{[H_3PO_4]}$$

$$pKa_2 = \frac{[HPO_4^{2-}][H_3O^+]}{[[H_2PO_4^-]]}$$

$$pKa_3 = \frac{[PO_4^{3-}][H_3O^+]}{[HPO_4^{2-}]}$$

The pH's of the solution at half neutralization points of the first second and third ionization stages are equal to pK_{a1}, pK_{a2}, pK_{a3} respectively.

PROCEDURE:

0.2 N phosphoric acid and 0.2 N NaOH is prepared respectively. 20 ml of the 0.2 N phosphoric acid is pipetted out into a clean SMF and made upto 100 ml. 20 ml of the made up solution is pipetted out into a clean beaker and 100 ml of water is added for dilution. The glass pH electrode is immersed in the solution after the calibration with buffer solutions of pH 4.7 and 9.3. The pH of the phosphoric acid is measured and then NaOH is added drop by drop, stirred continuously and the pH value is measured. The addition of NaOH is continued till the end points are reached. A graph is plotted between pH Vs. Volume of NaOH added.

RESULT:

a) pK_{a1} = _____ (first acid dissociation constant)

b) pK_{a2} = _____ (second acid dissociation constant)

c) pK_{a3} = _____ (Third acid dissociation constant)

INFERENCE:

The I,II, III order dissociation constant (pKa) of H_3PO_4 is calculated from the pH values.

NUMBER OF BENEFICIARIES:45 students per year

OUTCOME:

Students are able to measure pH of a solution using pH meter. They are able to calibrate the pH meter accurately. They are able to calculate the I,II, III order dissociation constant (pKa) of H_3PO_4 from the pH values.

2A. PHASE DIAGRAM – SIMPLE EUTECTIC SYSTEM

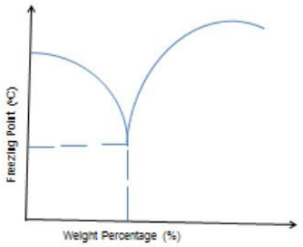
DETERMINATION OF FREEZING POINT A AND MIXTURE OF A AND B

Time in minutes	Pure A	Addition of B				
		I	II	III	IV	V

DETERMINATION OF FREEZING POINT B AND MIXTURE OF A AND B

Time in minutes	Pure B	Addition of A				
		I	II	III	IV	V

Weight A	Weight B	Weight percentage (%)	Freezing point (°C)



2A.PHASE DIAGRAM – SIMPLE EUTECTIC SYSTEM

AIM :

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

REQUIREMENTS:

EQUIPMENTS	: Weighing balance, sensitive thermometer, freezing test tube set, stop clock
CHEMICALS	: Components A(naphthalene) and B(biphenyl), weighing bottle
GLASSWARES	: Beaker 400ml, Conical flask

PRINCIPLE:

The simple phase diagram for a two component solid system represents the behaviour of the components which are completely soluble in each other when in the liquid state but immiscible in the solid phase. The temperature composition phase diagram for this equilibirium has the general form.

In the curve, a and b are the freezing points of 'A' and 'B'. Along the curve two phases are in equilibirium namely pure solid A with melt and pure solid B with melt. The two curves meet at the minimum point C. At the point C, three phases namely solid A, melt and solid B are in equilibirium and if has no degree of freedom. They are called eutectic temperature T_c and eutectic compositions X_c respectively.

PROCEDURE

The freezing tube set with a sensitive thermometer, stirrer and 5g of A is mounted in the water bath and heated. The heating is stopped when the solid melted completely to form a clear liquid. The tube taken out from the bath is introduced into an air jacket and allowed to cool steadily with stirring. The freezing point is found out from the appearance of first crystal while steady cooling. Now a weighed amount 1g of B was added to A and freezing point of this mixture is found out as before. Similarly the freezing point is found out for 4 more additions of B (each of 1g).

The same procedure is repeated by starting with 5g of B and adding to it 5 times 1g of A and finding out the freezing points of pure B and the resulting mixtures as above.

The results are tabulated. The freezing points are plotted against their corresponding composition. From the curve the eutectic temperature Tc and eutectic composition(X_c) are recorded.

RESULT:

Freezing point of pure A = (ºC)
Freezing point of pure B = (ºC)
Eutectic temperature (T_c) = (ºC)
Eutectic composition (X_c) = %

INFERENCE :Freezing point decreases with addition of other solute

NO. OF BENEFICIARIES:45

OUTCOME :

The students are able to construct the phase diagram for a binary mixture and to determine the eutectic temperature and the eutectic composition.

2B. PHASE DIAGRAM – COMPOUND FORMATION

DETERMINATION OF FREEZING POINT OF A MIXTURE OF A AND B

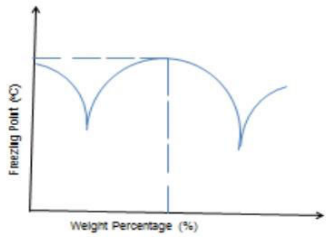
Time in sec	Pure A	Addition of B				
		1	2	3	4	5

DETERMINATION OF FREEZING POINT OF B MIXTURE OF A AND B

Time in sec	Pure B	Addition of A				
		1	2	3	4	5

Weight percentage of A = (weight of A/weight of A + weight of B) 100

Weight of A (g)	Weight of B (g)	Weight percentage (%)	Freezing point (°C)



2B. PHASE DIAGRAM – COMPOUND FORMATION

AIM :

To construct the phase diagram for the binary system forming a compound and to find the composition and temperature of the compound formation.

REQUIREMENTS :

EQUIPMENTS	: Weighing balance, sensitive thermometer, freezing test tube set, stop clock
CHEMICALS	: Compounds A(alpha naphthyl amine) and B(metadinitro benzene)
GLASSWARES	: Weighing bottle, beaker – 400 ml, conical flask

PRINCIPLE:

When the two compounds A and B react to form a compound AB stable upto its freezing point. The phase diagram has the appearance of two simple eutectics. In this phase diagram there are two univariant. Three phase equilibirium namely

i) Pure solid A melt compound AB

ii) Solid B melt compound AB at the equilibirium freezing point of the compound and the composition and temperature of the eutectics can be calculated.

PROCEDURE:

The freezing tube set with a sensitive thermometer, stirrer and 5g of A is mounted in the water bath and heated. The heating is stopped when the solid melted completely to form a clear liquid. The tube taken out from the bath is introduced into an air jacket and allowed to cool steadily with stirring. The freezing point is found out from the appearance of first crystal while steady cooling. Now a weighed amount 1g of B is added to A and freezing point of this mixture is found out as before. Similarly the freezing point is found out for 4 more additions of B (each of 1g).

The same procedure is repeated by starting with 5g of B and adding to it 5 times 1g of A and finding out the freezing points of pure B and the resulting mixtures as above.The freezing point of the pure component A is determined from the first crystal appearance while cooling. Five different solution of B in A are prepared by adding five portion of solute B to solvent A and the freezing point of the pure component B and the solvent B are determined.

The results are tabulated in a table and the freezing points are plotted against their composition. From graph the freezing point of the compound and the corresponding composition are tabulated.

RESULT:

Freezing point of pure A = (ºC)

Freezing point of pure B = (ºC)

Freezing point of compound AB = (ºC)

Composition of compound AB = (%)

INFERENCE: Freezing point decreases with addition of other solute

NO. OF BENEFICIARIES:45

OUTCOME:

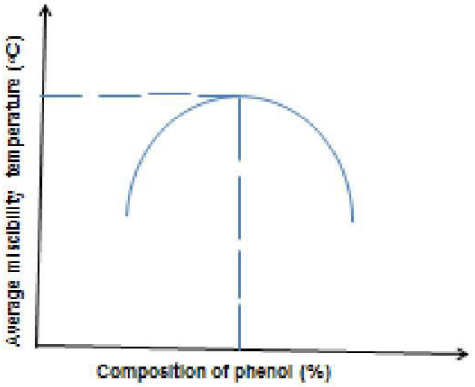
The students are able to construct the phase diagram for the binary system forming a compound and to find the composition and temperature of the compound formation

3. DETERMINATION OF CRITICAL SOLUTION TEMPERATURE (CST) OF PHENOL - WATER SYSTEM AND THE EFFECT OF IMPURITY ON CST

DETERMINATION OF THE CRITICAL SOLUTION TEMPERATURE OF THE PHENOL-WATER SYSTEM

Volume of Phenol (ml)	Volume of Water(ml)	Temperature at which turbidity disappears (°C)	Temperature at which turbidity appears (°C)	Average miscibility temperature (°C)

Volume of Phenol (ml)	Volume of Water(ml)	Temperature at which turbidity disappears (°C)	Temperature at which turbidity appears (°C)	Average miscibility temperature (°C)



3. DETERMINATION OF CRITICAL SOLUTION TEMPERATURE (CST) OF PHENOL - WATER SYSTEM AND THE EFFECT OF IMPURITY ON CST

AIM :

a) To determine the CST for a pair of partially miscible liquids phenol and water.

b) To study the effect of impurity on CST and to determine the concentration of the given electrolyte from miscibility temperature measurement.

REQUIREMENTS :

EQUIPMENTS	: Weighing balance, sensitive thermometer, boiling test tube set, stop clock
CHEMICALS	: Phenol and sodium chloride
GLASSWARES	: Weighing bottle, beaker – 400 ml, conical flask, graduated pipette, SMF

PRINCIPLE :

When two partially miscible liquids are mixed, they form two immiscible layers existing in equilibirium. The upper layer is the solution of water in phenol and lower layer is the solution of phenol in water. As the temperature is raised the mutual solubility of two liquids increases and at a particular temperature they are completely miscible forming a mixture. This temperature is called miscibility temperature. However such a system becomes completely miscible into a homogeneous mixture above a certain temperature. This temperature (CST) is calledconsolute temperature.

The CST of phenol-water system is greatly influenced by the addition of an electrolyte. The mutual solubility of phenol and water are affected in the presence of an electrolyte. If a substance is a capable of dissolving in only one liquid, it results in the increase of miscibility temperature and hence alters the CST of the system. A substance dissolving in both the layers totally decreases the CST of the system. This effect has been used to determine the concentration of a given electrolyte solution.

PROCEDURE:

Accurately 5 ml of freshly distilled phenol was taken in a boiling tube fitted with a cork carrying a sensitive thermometer and stirrer. Accurately 1 ml of distilled water was added with the help of graduated pipette. The boiling tube was kept in a waterbath. The waterbath was gradually heated at which temperature the two liquids become completely miscible was noted. The boiling tube was allowed to cool in an air-jacket with constant stirring and the temperature at which turbidity first appears was noted. The average of these two temperature was the miscibility temperature for that composition.

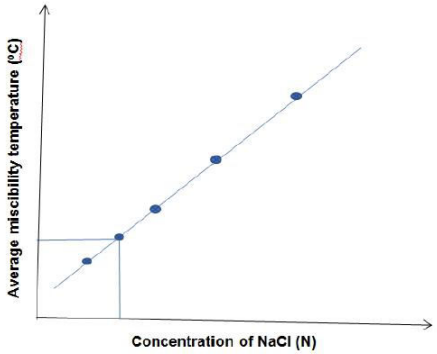
The experiment was repeated by adding 1 ml of water each time and their corresponding percentage composition of phenol was calculated. The miscibility temperature were then plotted against weight percentage composition of phenol. A graph of parabolic shape is obtained.

The maximum of the above curve indicates the CST of phenol-water system.

3. DETERMINATION OF CRITICAL SOLUTION TEMPERATURE (CST) OF PHENOL - WATER SYSTEM AND THE EFFECT OF IMPURITY ON CST

EFFECT OF IMPURITY ON CST AND DETERMINATION OF STRENGTH OF GIVEN NaCl SOLUTION

Strength of NaCl Solution (N)	Temperature at which turbidity disappears (°C)	Temperature at which turbidity appears (°C)	Average miscibility temperature (°C)
0.02			
0.04			
0.06			
0.08			
unknown			



3. DETERMINATION OF CRITICAL SOLUTION TEMPERATURE (CST) OF PHENOL - WATER SYSTEM AND THE EFFECT OF IMPURITY ON CST

EFFECT OF IMPURITY ON CST AND DETERMINATION OF STRENGTH OF THE GIVEN NaCl SOLUTION:

250 ml of 0.1N NaCl was prepared in a SMF by dissolving 1.46g of NaCl. Then solutions of 0.02N, 0.04N, 0.06N, 0.08N were prepared from the above solution by appropriate dilution.

5 ml of phenol and 5 ml of 0.1N NaCl were taken in a boiling tube along with a stirrer and a thermometer. The tube was heated gently till turbidity disappears and a homogeneous transparent liquid phase was obtained. The miscibility temperature was noted. The temperature at which turbidity appears was also noted. The average of the two temperature was calculated.

The experiment was repeated with NaCl of different concentrations. Similarly average miscibility temperature of the given unknown NaCl was also found out. A graph was drawn with average miscibility temperature versus concentration. It is possible to determine the strength of the given NaCl solution by knowing its average miscibility temperature.

RESULT:

Critical solution temperature of phenol – water system = ^{O}C

The composition of phenol at critical solution temperature = %

Normality of the given NaCl = N

INFERENCE:

On increasing the concentration of the impurity, the average miscibility temperature increases, hence a straight line is obtained.

NO. OF BENEFICIARIES: 43

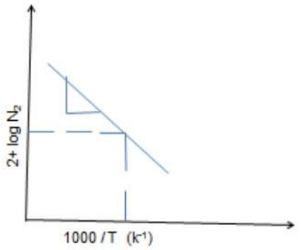
OUTCOME:

Students are able to determine theCST for a pair of partially miscible liquids phenol and water and to understand the effect of impurity on CST and to determine the concentration of the given electrolyte from miscibility temperature measurement.

4. ENTHALPHY OF SOLUTION AMMONIUM OXALATE IN WATER SYSTEM

TABLE 1 :

S.No	Weight of ammonium oxalate (g)	Volume of water (ml)	Saturation temperature (°C)
1	2.001	8	
2	2.001	9	
3	2.001	10	



ENTHALPY CALCULATION:

ΔH=slope X 2.303 X 8.314 KJ/mole

S.No	Weight of ammonium oxalate(g)	Volume of the solvent (ml)	Saturation temperature(o0C)	Temperature	1000/T lnk	N_2	log N_2	2+logN2

4. ENTHALPHY OF SOLUTION AMMONIUM OXALATE IN WATER SYSTEM

AIM:

To determine the heat of solution of the given solid A(ammonium oxalate) in the liquid solvent B (water) and determine its composition in the given solution.

REQUIREMENTS:

EQUIPMENTS	: Weighing balance, sensitive thermometer, boiling test tube set.
CHEMICALS	: Ammonium oxalate
GLASSWARES	: Weighing bottle, beaker – 400 ml, pipette, Standard Measuring Flask

PRINCIPLE:

In the binary solution, the compound in large amount is called the solvent and that present in small amount is called solute.It is known that at the saturation point the solid state at equilibrium with the solution.By finding out the saturation point for different compositions of the solution, the solution enthalpy can be easily determined.

$$N_2 = w_2/m_2/(w_1/m_1 + w_2/m_2)$$

PROCEDURE:

About 2 g of ammonium oxalate is taken in a boiling tube and place a thermometer and a stirrer in it and then add 7 ml of water from burette. Immerse the boiling tube in a beaker of warm water and stirred until all the solid dissolved, after thatremove the boiling tube from warm water.

Note the temperature at which the first crystals saturated and further 0.2 ml portion of water is added, repeat the same procedure to give new saturated temperature. Report the saturation temperature every time.

From the calculated volume, the graph is drawn between 1000/T and 2+ log N_2. The straight line is obtained .By using the data ΔH_3 can be calculated from the slope. From the graph we can calculate the weight of the given solute.

RESULT:

(i) The enthalpy of the solution ammonium oxalate in water = 10.9406 KJ/mol
(ii) The weight of unknown solute = 2.1122 g

INFERENCE:

On plotting 2+ log N_2Vs1000/T , a straight line with negative slope is obtained.

OUTCOME:

Students are able to determine the heat of solution of the given solid A(ammonium oxalate) in the liquid solvent B (water) and determine the composition of the given solution

5A. DETERMINATION OF PARTITION COEFFICIENT OF IODINE BETWEEN H_2O AND CCL_4, EQUILIBIRIUM CONSTANT OF THE REACTION BETWEEN KI AND IODINE

STANDARDIZATION OF THIO USING STANDARD$K_2Cr_2O_7$

S. No	Volume of $K_2Cr_2O_7$ (ml)	Burette reading (ml)		Volume of thio (ml)	Concordant value (ml)	Indicator
		Initial	Final			

DISTRIBUTION OF I_2 BETWEEN H_2O AND CCl_4

Bottle	Volume of organic layer(ml)	Burette reading (ml)		Volume of thio (ml)	Volume of aqueous layer (ml)	Burette reading (ml)		Volume of thio (ml)
		Initial	Final			Initial	Final	
A								
B								

DISTRIBUTION OF I_2 BETWEEN KI AND CCl_4

Bottle	Volume of organic layer(ml)	Burette reading (ml)		Volume of thio (ml)	Volume of aqueous layer (ml)	Burette reading (ml)		Volume of thio (ml)
		Initial	Final			Initial	Final	
C								
D								

5A. DETERMINATION OF PARTITION COEFFICIENT OF IODINE BETWEEN H_2O AND CCl_4, EQUILIBIRIUM CONSTANT OF THE REACTION BETWEEN KI AND IODINE

AIM :

To determine the partition co-efficient of iodine between CCl_4 and water

To determine the equilibrium constant of the reaction KI + I_2 $\rightleftharpoons$ KI_3 by the distribution method.

REQUIREMENTS:

EQUIPMENTS	Weighing balance
CHEMICALS	Saturated solution of I_2 in CCl_4, 10% KI solution, solid potassium dichromate, N/20 sodium thiosulphate solution
GLASSWARES	Weighing bottle, Burette, stoppered bottles, conical flask, Measuring cylinder – 50ml, 10ml, pipette – 25ml and 5ml, standard measuring flasks – 250ml and 100ml

PRINCIPLE:

When a solute like I_2 is added to a mixture of two immiscible liquids, iodine distributes itself between the liquid in such a way that the ratio of the concentration of I_2 in the two layers is constant. This ratio is called partition or distribution co-efficient. It is independent of the amount of the substance added to the liquid pair.

The partition or distribution constant is calculated using the formula

$$K_D = \frac{\text{conc. of } I_2 \text{ in organic layer}}{\text{Conc. of } I_2 \text{ in aqueous layer}}$$

$$K_D = \frac{[I_2]_{org}}{[I_2]_{aq}}$$

When I_2 is distributed between KI solution and CCl_4, a part of iodine is combined with KI to form I_3^-. The reaction can be written as,

$$KI + I_2 \rightleftharpoons KI_3$$

The equilibrium constant,

$$k = C_{KI3} / C_{KI} \times C_{I_2}$$

C_{KI3} is the concentration of triiodide formed.C_{KI} and C_{I2} are the concentration of KI and iodine at equilibrium in aqueous layer.

PROCEDURE:

A) DETERMINATION OF PARTITION COEFFICIENT OF IODINE BETWEEN CCL_4 AND H_2O:

The following volumes of liquid were carefully measured out into two 250ml stoppered bottles.

Contents	Bottle A	Bottle B
H_2O	100 ml	100 ml
I_2 in CCl_4	15 ml	10 ml
CCl_4	5 ml	10 ml

The bottles were shaken in a mechanical shaker for 30minutes. Then they were allowed to stand for some time in a trough containing water till the two layers separate. 5 ml of CCl_4 layer of the each bottle was pipetted out, added 10 ml of 10% KI , 15 ml of distilled water and titrated against N/20 sodium thiosulphate solution taken in a burette using starch as an indicator. The end point was the disappearance of blue colour.

25 ml of the aqueous layer of each bottle was pipetted out, added 10 ml of 10% KI, 15 ml of distilled water and titrated against N/200 sodium thiosulphate solution which has been prepared by diluting 10 ml of N/20 $Na_2S_2O_3$ in a 100ml standard measuring flask. The end point was the disappearance of blue colour. The titrations were duplicated. The distribution or partition coefficient was then calculated from the titre values.

B) DETERMINATION OF EQUILIBRIUM CONSTANT:

250 ml of N/10 KI solution was prepared in a standard flask by dissolving about 4.2g of KI (equivalent weight of 166).Using burette and pipette the following volumes were measured into 250 ml stoppered bottles.

Contents	Bottle C	Bottle D
KI solution	100 ml	100 ml
I_2 in CCl_4	15 ml	10 ml
CCl_4	5 ml	10 ml

The contents of the bottles were shaken well for about one hour to attain equilibrium. The bottles were kept in a constant temperature bath till the two layers separate out. 5 ml of CCl_4 layer of each bottle was pipetted out, added 10 ml of 10% KI, 15 ml of distilled water and this was titrated against N/20 $Na_2S_2O_3$ solution using starch as indicator. The end point was the disappearance of blue colour. 25 ml of the aqueous layer of each bottle is pipetted out ,added 10 ml of 10% KI, 15ml distilled water and titrated against N/20 $Na_2S_2O_3$ solution using starch as indicator. The end point was the disappearance of blue colour. The titrations were duplicated.

The given $Na_2S_2O_3$ solution was standardized by preparing 0.1N $K_2Cr_2O_7$ solution. 100 ml of 0.1N $K_2Cr_2O_7$ was prepared by dissolving about 0.5g (equivalent weight of $K_2Cr_2O_7$ = 49) of it in a 100 ml standard measuring flask. 10ml of $K_2Cr_2O_7$ was pipetted out into a clean conical flask, added about 10ml of 10% KI and 5ml of 2N HCl and titrated the liberated I_2 against $Na_2S_2O_3$ taken in the burette. When the solution was pale yellow added 1ml of starch and continued the titration till the colour changes from blue to green. The titration was repeated to get concordant values and calculated the strength of $Na_2S_2O_3$. The equilibirium constant was calculated using the titre values.

RESULT:

i) Partition coefficient of iodine between water and CCl_4:

Bottle A: K_D =

Bottle B : K_D =

ii) Equilibirium constant of the reaction, $KI + I_2 \rightleftharpoons KI_3$

Bottle C

K_{eq} = i)

ii)

Bottle D

K_{eq}= i)

ii)

INFERENCE: Iodine is distributed between two immiscible liquids to a constant extend.

NO. OF BENEFICIARIES:45

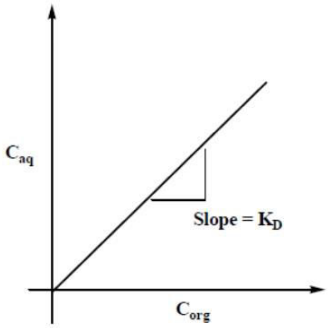
OUTCOME: Students are able to distribute iodine between two immiscible liquids and to determine the partition coefficient and equilibrium constant.

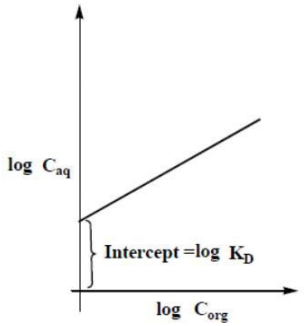
5B. DETERMINATION OF PARTITION COEFFICIENT OF BENZOIC ACID IN BENZENE AND WATER

PARTITION COEFFICIENT OF BENZOIC ACID IN BENZENE AND WATER

Bottle No	V_{org}	Vaq	$N_{org}=C_{org}$	$N_{aq}=C_{aq}$	K	log C_{org}	log C_{aq}
1							
2							
3							

S. No	Volume of NaOH(ml)	Burette reading		Concordant Value (ml)	Indicator
		Initial (ml)	Final (ml)		





5B. DETERMINATION OF PARTITION COEFFICIENT OF BENZOIC ACID IN BENZENE AND WATER

AIM:

To determine the partition coefficient of benzoic acid between benzene and water.

REQUIREMENTS:

EQUIPMENTS	Weighing balance
CHEMICALS	Benzoic acid, benzene, phenolphthalein, NaOHsolution
GLASSWARES	Weighing bottle, Burette, stoppered bottles, conical flask, Measuring cylinder – 50ml, 10ml, pipette – 25ml and 5ml, standard measuring flasks – 250ml and 100ml

PRINCIPLE:

When a solute is shaken with two immiscible solvents, it gets distribute between the solvents. This distribution of solute in two solvents depends upon the solubility of the solute in two solvents is constant at a given temperature. The constant is called the partition coefficient (K) or the distribution solvents.

PROCEDURE:

Prepare the following mixtures.

Set – I

25 ml water + 25 ml of saturated, solution of benzoic acid in benzene.

Set – II

25 ml of water + 20 ml saturated solution Benzoic acid in Benzene + 5 ml benzene.

Set –III

25 ml of H_2O + 15 ml saturated solution of Benzoic acid in benzene + 10 ml benzene shaken the mixtures vigorously for about 1 hour. So that the benzoic acid gets distributed between the two solvents and the distribution equilibrium is reached. Allow the bottles to stand for 10 minutes to separate into two clean layers. (lower layer – aqueous layer).

Pipette out 5 ml of organic layer (benzene layer) into a conical flask containing 10 ml of water and titrate against 0.1 N NaOH using phenolphthalein as an indicator. End point is appearance of pink colour. Pipette out 10 ml of aqueous layer and add 1 drop of

phenolphthalein indicator and titrate it against 0.01 N NaOH solution. End point is the appearance of pinkcolour.

Where,

V_{org} – volume in ml of 0.1N NaOH per 5 ml of orgainic layer.

V_{aq} – volume of 0.01N NaOH per 10 ml of aquous layer.

N_{org} – normality of organic layer.

N_{aq} – normality of aqueous layer.

C_{org} – concentration of organic layer in g mole litre normality (N_{org}).

C_{aq} – concentration of aqueous layer in g mol / in normality (N_{aq})

$K = \frac{C_{aq}}{C_{org}^{1/2}}$ partition coefficient of benzoic acid in water and benzene.

RESULT:

K_D (From calculations) = ……

K_D value from Graph -1 =……

K_D value from Graph - 2 = ……..

INFERENCE:

On plotting C_{aq}VsC_{org}, Straight line passing through origin is obtained with positive slope.Thus Nernst Distribution law is verified.

NO. OF BENEFICIARIES:45

OUTCOME:

Students were able to verify the Nernst Distribution and to determine the partition coefficient.

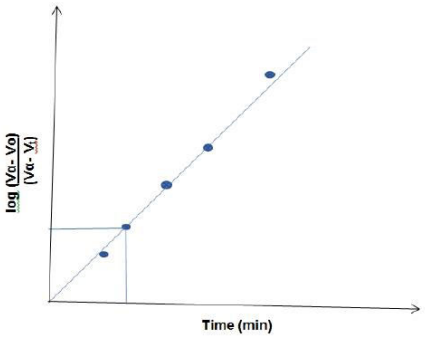
6A.DETERMINATION OF RELATIVE STRENGTH OF ACIDS BY KINETIC STUDY OF HYDROLYSIS

HYDROLYSIS OF ESTER USING ACID A

Time in (mins)	Burette reading (ml)		Volume of NaOH (ml)	log (Vα- Vo) / (Vα- V_t)	K= (2.303/t) log (Vα- Vo) / (Vα- V_t)
	Initial	Final			

HYDROLYSIS OF ESTER USING ACID B

Time in (mins)	Burette reading (ml)		Volume of NaOH (ml)	log (Vα- Vo) / (Vα- V_t)	K= (2.303/t) log (Vα- Vo) / (Vα- V_t)
	Initial	Final			



6A.DETERMINATION OF RELATIVE STRENGTH OF ACIDS BY KINETIC STUDY OF HYDROLYSIS

AIM:

To study the kinetics of acid catalysed hydrolysis of the given ester and to compare the strength of the two given acids.

REQUIREMENTS:

EQUIPMENTS	: Weighing balance,stop clock, water bath
CHEMICALS	:Ester, acid, sodium hydroxide, Phenolphthalein
GLASSWARES	:Weighing bottle, burette, stoppered bottles, conical flask, pipette – 20ml standard measuring flasks –100ml

PRINCIPLE:

The hydrolysis of ester is a reversible reaction

$$CH_3COOC_2H_5 + H_2O \underset{\leftarrow}{\rightarrow} CH_3COOH + C_2H_5OH$$

In the presence of excess water, the reaction proceeds almost to completion in the forward direction. It is a pseudo unimolecular reaction and the following rate equation is applicable to the above reaction.

$$\text{Rate} \propto [\text{ester}]\,[H_2O]$$

$$-dC/dt = k[\text{ester}]\,[H_2O] \text{ since } [H_2O] \text{ remains constant}$$

$$-dC/dt = k^1[\text{ester}] \text{ where } k^1 = K[H_2O]$$

k is the specific velocity constant, 'C' is the concentration of ester at time t During hydrolysis, acetic acid is produced and concentration of acetic acid increases as the reaction progresses. Hence the disappearance of ester concentration can be followed by drawing a known volume of the mixture at regular intervals and titrating the reaction mixture against NaOH solution.

PROCEDURE:

Exactly 100ml of the given acid A was taken in a 250 ml clean stoppered bottle. Acid A and the given ester were kept in the water bath for 10 minutes to obtain the steady temperature . When the solutions have attained the steady temperature, 5 ml of ester was pipetted out into acid A. zero time was noted by starting a stop clock, when the half of the volume of the ester solution was transferred into the reaction bottle. After thorough mixing, 5ml of the solution was pipetted out into a clean conical flask containing few piece of

ice. It was then titrated against NaOH solution taken in the burette using phenolpthalein indicator. The end point was the appearance of pale pink colour. The titre volume was taken as V_o. Similarly 5 ml of the reaction mixture was withdrawn at regular intervals 5, 10, 15, 20, 25, 30, 35,40, 45, 50,55,60 minutes and titrated as before. The titre value V_t was noted. In the middle of the experiment ,20ml of mixture was taken in a 250ml conical flask closed with a rubber cork and refluxed over a water bath for about 45 minutes. This was cooled and 5 ml of the reaction mixture was pipetted out into conical flask and titrated. The titre value corresponds to the completion of the reaction and it was taken as V_∞. The same procedure was repeated with acid B.

RESULT:

1) The rate constant of the hydrolysis of ester using acid A

i) From graph , k_A =
ii) From experiment, k_A =

2) The rate constant of the hydrolysis of ester using acid B

i) From graph , k_B=
ii) From experiment, k_B=

3) The relative strength of two given acids
i) From graph, k_A / k_B =
ii) From experiment, k_A / k_B =

INFERENCE: log $[(V_\alpha - V_o)/(V_\alpha - V_t)]$ varies linearly with time.

NO. OF BENEFICIARIES: 43

OUTCOME:

Students are ableto study the kinetics of acid catalysed hydrolysis of the given ester and to compare the strength of the two given acids.

6B. ANALYSIS OF DYE DEGRADATION USING UV PHOTO REACTOR

CALCULATION

% Degradation = $C_0 - C / C_0 \times 100$

Or

% Degradation = $A_0 - A / A_0 \times 100$

λ_{max}=661 nm

Concentration of dye(ppm)	Absorbance
20	
40	
60	
80	
100	

6B. ANALYSIS OF DYE DEGRADATION USING UV PHOTO REACTOR

AIM:

To determine the rate of dye degradation using photoreactor.

REQUIREMENTS:

EQUIPMENTS	:Weighing balance, Spectrophotometer UVphotoreactor
CHEMICALS	TiO_2, methylene blue dye
GLASSWARES	:Weighing bottle, conical flask, pipette, standard measuring flasks –100ml

PRINCIPLE:

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

$$A = \log(I_0/I_t) = \varepsilon c l$$

where,

$\log(I_0/I_t)$is the optical density at λ_{max}
ε is the molar extinction of co-efficient
c is the concentration of the sample
l is the path length of the sample in cm

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

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

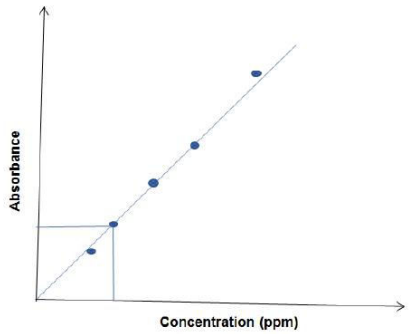
A linear relation exists between absorbance (O. D) and concentration of the sample is known as Beer's law.

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

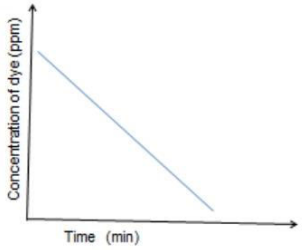
Before verification of Beer's law, it is necessary to a select a suitable wave length and determines whether Beer's law is valid at the wavelength selected. The most suitable wavelength is that at which maximum absorbance is observed, called λ_{max}. The λ_{max} will always be at the same wavelength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

6B. ANALYSIS OF DYE DEGRADATION USING UV PHOTO REACTOR

CALIBRATION GRAPH



Time (min)	Absorbance	Concentration of dye(ppm)
0		
5		
10		
15		
20		



6B. ANALYSIS OF DYE DEGRADATION USING UV PHOTO REACTOR

PROCEDURE:

100 mg of methylene blue dye powder is accurately dissolved in 1000ml of distilled water. First of all, a 100 mg precisely weighted dye powder was allowed to dissolve in 200ml double distilled water by shaking thoroughly. After complete dissolution of dye it was transferred in a volumetric flask (1000ml) then double distilled water was added up to the 1 liter mark of volumetric flask. A stock solution prepared was 100 mg/liter (100 ppm) with double distilled water. By using this stock solution, next low concentration was prepared (80,60,40,20 ppm) by subsequent dilution. The optical density of the supernatant dye solution was measured at wavelength of 661 nm by using JEOL UV-Visible spectrophotometer. A calibration curve was drawn by plotting optical density (y-axis) against concentration (x-axis).A straight line passing through origin was obtained. Molar extinction coefficient can be determined from the slope of the line.

The photo-degradation of methylene blue dye was studied in a simple UV photo-reactor. Exactly 0.5g of TiO_2photocatalyst was weighed accurately and to it added 10 ml of methylene blue dye solution (100 ppm), stirred well and kept in the UV photoreactor under the irradiation of UV light. For every 5 minutes, an aliquot of sample was taken from the reaction mixture and its optical density was measured. This was repeated for every 5 minutes until the dye becomes colourless (Concentration becomes zero). As the absorbance or optical density of MB solution used in this experiment is directly proportional to its concentration (C), the concentration of the dye after time t minutes was known from the calibration graph. Then a graph was plotted between concentration of dye Vs time. From that time taken for complete degradation dye was noted. % degration of the dye at particular time t was calculated from the formula.

RESULT:

Time take for complete degradation of dye is min.

% Degradation of dye at time t min is **%**

INFERENCE:

As Concentration increases, absorbance increases. Hence the straight line plot is obtained.

NO. OF BENEFICIARIES: 43

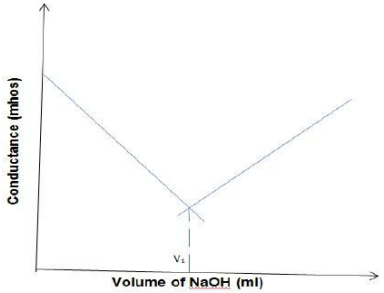
OUTCOME:

Students are able to determine the time required for the complete degradation of dye and to calculate the % of dye degradation at any time.

7A.CONDUCTOMETRIC TITRATION

STANDARDISATION OF HCL (STANDARD NAOHVS UNKNOWN HCL

S.No	Volume of NaOH (ml)	Conductance (mhos)



CALCULATION

Volume of NaOH (V_1) = V_1 ml

Normality of NaOH (V_1) = y N

Volume of HCl (V_2) = 40 ml

Normality of HCl (N_2) = (V_1 x y/40) N

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7A.CONDUCTOMETRIC TITRATION

Conductometric titration between an acid and a base (HClVsNaOH)

AIM:

To estimate the concentration of the given acid by titration against standard alkali conductometrically.

REQUIREMENTS:

EQUIPMENTS	: Weighing balance,Digital Conductivity Meter, Conductivity Cell,
CHEMICALS	:Hydrochloric acid and sodium hydroxide
GLASSWARES	: Weighing bottle, microburette, pipette – 20ml standard measuring flasks –100ml

PRINCIPLE:

When a strong alkali is added to an acid the conductance of the solution will decrease. This occurs due to the disappearance of the hydrogen ion and its replacement by less mobile ions Na^+ of the base. When all the hydrogen ions in acid is neutralised by hydroxide ion from the base added, then further addition of alkali will lead to increase in conductance due to the presence of hydroxyl ions. The conductance of the solution is plotted against the volume of alkali added. The point of intersection of the two lines gives the volume of alkali required to neutralize the acid.

PROCEDURE:

A known volume (40 ml) of the acid was pipetted out into a clean 100ml beaker. A clean conductivity cell was introduced into the solutions in the beaker and it was connected to digital conductivity meter. The range switch in DCM was kept in extreme clockwise position. The DCM was switched ON and Kc, the specific conductivity of the solution displaced on the digital panel meter (DPM) was noted.

The alkali was taken in a clean rinsed micro burette. The burette was mounted above the liquid. The alkali was added in 1 ml portions, stirred well. Carefully, using the cell the conductivity was noted in DPM. This pilot titration fixes the equivalence point. The cell was carefully taken from the solution, washed and rinsed with distilled water. The actual titration was done by pipetting out 40 ml of the standard acid in the clean 100ml beaker adding 1 ml of NaOH, stirred well and noted the conductance. A graph was drawn plotting conductance against volume of the titrant (NaOH) added the point of intersection of 2 lines

gives the equivalent point. By knowing the strength of NaOH, the strength of the acid was calculated.

RESULT:

Normality of the given HCl = N

INFERENCE:

On adding NaOH to HCl, the value of conductance decreases before the neutralisation point and then begins to increase.

NO. OF BENEFICIARIES:43

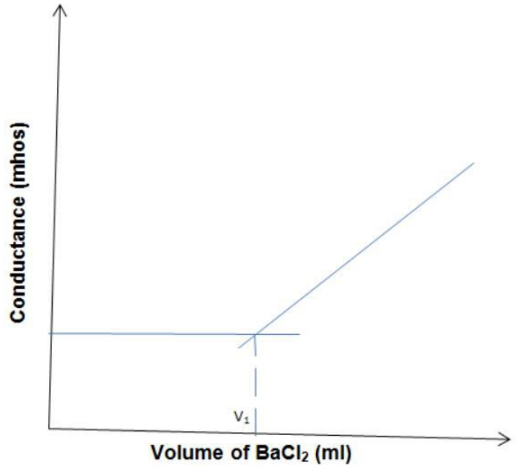
OUTCOME:

Students are able to handle the conductivity meter and conductivity cell and they are also able to determine the normality of the given HClconductometrically.

7B.CONDUCTOMETRIC PRECIPITATION TITRATION ($BaCl_2$ VS K_2SO_4)

STANDARDISATION OF K_2SO_4 (STANDARD $BaCl_2$VS UNKNOWNK_2SO_4)

S.No	Volume of NaOH (ml)	Conductance (mhos)



CALCULATION

Volume of $BaCl_2$ (V_1) = V_1 ml

Normality of $BaCl_2$ (V_1) = y N

Volume of K_2SO_4 (V_2) = 40 ml

Normality of K_2SO_4 (N_2) = (V_1 x y/40) N

7B.CONDUCTOMETRIC PRECIPITATION TITRATION ($BACl_2$ VS K_2SO_4)

AIM :

To estimate the concentration of the given K_2SO_4 by titration against standard $BaCl_2$ conductometrically.

REQUIREMENTS:

EQUIPMENTS	:Weighing balance, Digital Conductivity Meter, conductivity cell,
CHEMICALS	:barium chloride solution, potassium sulphate solution.
GLASSWARES	:Weighing bottle, microburette, pipette – 20ml standard measuring flasks –100ml

PRINCIPLE :

When a solution of K_2SO_4 is treated against $BaCl_2$, precipitate of $BaSO_4$ is formed.

$K_2SO_4 + BaCl_2 \longrightarrow BaSO_4 + 2KCl$

The conductance of the solution does not change since there is no difference in conductance between $BaCl_2$and K_2SO_4. The conductance remains almost constant until the equivalence point. After the complete precipitation of the sulphate ion, further addition of $BaCl_2$ increase the conductance of the solution sharply.The conductance of the solution is plotted against the volume of $BaCl_2$ added. The point of intersection of the two lines gives the volume of $BaCl_2$ is required for complete precipitation of K_2SO_4.

PROCEDURE:

Accurately 40 ml of the given K_2SO_4 was pipetted out into a clean 100ml beaker. A clean conductivity cell was introduced into the solution in the beaker and it is connected to digital conductivity meter. The range switch in DCM was kept in extreme clockwise position. The DCM was switched ON and Kc, the specific conductivity of the solution displaced on the digital panel meter (DPM) was noted.

Standard $BaCl_2$ solution was taken in a clean rinsed micro burette. The burette was mounted above the liquid. The $BaCl_2$ is added in 1 ml portions, stirred well. Carefully using the cell, the conductivity is noted in DPM. This pilot titration fixes the equivalence point. The cell is carefully taken from the solution, washed and rinsed with distilled water. The actual titration is done by pipetting out 40 ml of the standard acid in the clean 100ml beaker adding 1 ml short of the range fixed in the pilot experiment, stirred well and noted the

conductivity. A graph is drawn plotting conductance against volume of the $BaCl_2$ added the point of intersection of 2 lines gives the equivalent point. Knowing the strength of $BaCl_2$, the strength of the K_2SO_4 is calculated.

RESULT:

Normality of the given K_2SO_4 = N

INFERENCE:

On adding $BaCl_2$ to K_2SO_4, the value of conductance remains constant before the equivalence point and then begins to increase.

NO. OF BENEFICIARIES:43

OUTCOME:

Students are able to handle the conductivity meter and conductivity cell and to determine the normality of the given K_2SO_4conductometrically.

8A. POTENTIOMETRIC REDOX TITRATIONS ($KMnO_4$ VS FAS)

TRIAL EXPERIMENT

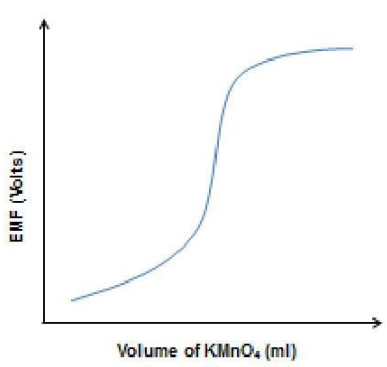
$KMnO_4$ Vs FAS

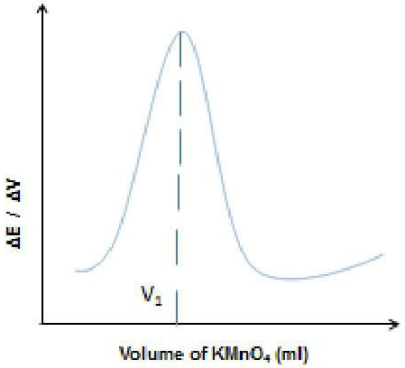
Volume of $KMnO_4$ (ml)	EMF in volts

ACTUAL EXPERIMENT

$KMnO_4$ Vs FAS

Volume of $KMnO_4$ (ml)	EMF (Volts)	ΔE in Volts	ΔV in (ml)	ΔE / ΔV





8A.POTENTIOMETRIC REDOX TITRATIONS ($KMnO_4$ VS FAS)

AIM :

To titrate Ferrous Ammonium Sulphate (FAS) potentiometrically with standard Potassium permanganate ($KMnO_4$) and to determine the strength of FAS solution.

REQUIREMENTS:

EQUIPMENTS	:Weighing balance,digital voltmeter, calomel electrode, platinum electrode
CHEMICALS	:Ferrous ammonium sulphate, potassium permanganate
GLASSWARES	:Weighing bottle, microburette, pipette – 20ml standard measuring flasks –100ml

PRINCIPLE :

$KMnO_4$oxidises the ferrous ions into ferric ions in the presence of acid. The reaction involved is

$$5Fe^{2+} + KMnO_4 + 8H^+ \longrightarrow 5Fe^{3+} + Mn^{2+} + 4H_2O$$

The electrode potential in this titration depends upon the concentration of Fe^{2+}, Fe^{3+}, and H^+ ion concentration on electrode potential, the titration was usually carried out in the presence of large excess of an acid. The oxidation potential of this redox system was given by,

$$E = E^{\circ} - \frac{RT}{F} \ln \frac{(Fe^{3+})}{(Fe^{2+})}$$

The necessary cell is set up by connecting the redox electrode against a calomel electrode as shown below

$$Pt/Hg/HgCl_{2(solution)}/\ Kcl_{(std)}\ //\ Fe^{2+}\ ,\ Fe^{3+}/Pt$$

When $KMnO_4$ was added to the system, Fe^{2+} is converted into Fe^{3+} whose concentration increases with the progressive addition of $KMnO_4$ and observed cmf will gradually increase. At the end point there will be a sharp change due to sudden removal of all Fe^{2+} ions. A plot between emf measured Ecell or E/V against volume of $KMnO_4$ added was drawn and the end point is determined.

8A.POTENTIOMETRIC REDOX TITRATIONS ($KMnO_4$ VS FAS)

CALCULATION

Volume of $KMnO_4$ (V_1) = V_1 ml

Normality of $KMnO_4$ (V_1) = y N

Volume of FAS (V_2) = 20 ml

Normality of FAS (N_2) = (V_1 x y/20) N

8A.POTENTIOMETRIC REDOX TITRATIONS ($KMnO_4$ VS FAS)

PROCEDURE :

20 ml of FAS was pipetted out into a 250ml beaker and an equal volume of 2N sulphuric acid was added to it. A clean platinum electrode was introduced into the solution. It was connected to a calomel electrode through KCl salt bridge. The two electrodes were connected to a digital voltmeter to get a positive emf value. $KMnO_4$ solution was taken in a clean burette. The burette was so maintained above the beakers that its tip was just above the liquid. The trial experiment was done by adding 1 ml portion at a time noting the emf value for each addition. This trial titration gave the equivalence point within 1 ml range. Now another 20 ml of FAS solution was pipetted out into a clean beaker added an equal volume of 2N H_2SO_4 and the electrodes were introduced as before. $KMnO_4$ solution was added from the burette in bulk till its volume 1 ml short of the range fixed in the previous trial experiment. The solution was stirred well and the emf of the cell was measured. Now 0.1 ml portion of the titrant was added at a time and for each addition emf was noted.

A graph was plotted againstthe volume of $KMnO_4$ added and emf. The accurate end point was determined by plotting E/V Vs volume of $KMnO_4$ added. From the end point, strength of FAS solution is calculated.

RESULT :

Normality of FAS = N

INFERENCE:

Rise in EMF is high nearer to the equivalence point and then begins to decrease

NO. OF BENEFICIARIES:43

OUTCOME:

Students are able to construct the electrochemical cell, operate the potentiometer and to determine the normality of the given FAS potentiometrically.

8B.POTENTIOMETRIC REDOX TITRATIONS ($K_2Cr_2O_7$ VS FAS)

TRIAL EXPERIMENT

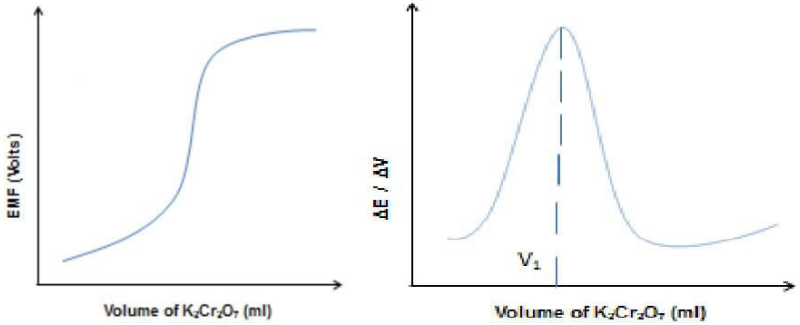
$K_2Cr_2O_7$ Vs FAS

Volume of $K_2Cr_2O_7$ (ml)	EMF in volts

ACTUAL EXPERIMENT

$K_2Cr_2O_7$ Vs FAS

Volume of $K_2Cr_2O_7$(ml)	EMF (volts)	ΔE in volts	ΔV in (ml)	ΔE / ΔV



8B.POTENTIOMETRIC REDOX TITRATIONS ($K_2Cr_2O_7$ VS FAS)

AIM :

To titrate Ferrous Ammonium Sulphate (FAS) potentiometrically with standard $K_2Cr_2O_7$ and to determine the strength of FAS solution.

REQUIREMENTS:

EQUIPMENTS	:Weighing balance,digital voltmeter, calomel electrode, platinum electrode
CHEMICALS	:Ferrous ammonium sulphate, potassium dichromate
GLASSWARES	:Weighing bottle, microburette, pipette – 20ml standard measuring flasks –100ml

PRINCIPLE :

$K_2Cr_2O_7$oxidises the ferrous ions into ferric ions in the presence of acid. The reaction involved is

$$6Fe^{2+} + K_2Cr_2O_7 + 14H^+ \longrightarrow 6Fe^{3+} + 2Cr^{3+} + 7H_2O$$

The electrode potential in this titration depends upon the concentration of Fe^{2+}, Fe^{3+}, and H^+ ion concentration on electrode potential, the titration was usually carried out in the presence of excess of an acid. The oxidation potential of this redox system was given by,

$$E = E^{o}- \frac{RT}{F} \ln \frac{(Fe^{3+})}{(Fe^{2+})}$$

The necessary cell was set up by connecting the redox electrode against a calomel electrode as shown below

$$Pt/Hg/HgCl_{2(solution)}/ Kcl_{(std)} // Fe^{2+}, Fe^{3+}/Pt$$

When $K_2Cr_2O_7$ was added to the system, Fe^{2+} is converted into Fe^{3+} whose concentration increases with the progressive addition of $K_2Cr_2O_7$ and observed emf will gradually increase. At the end point there will be a sharp change due to sudden removal of all Fe^{2+} ions. A plot drawn between emf measured Ecell or E/V against volume of $KMnO_4$ added and the end point was determined.

8B. POTENTIOMETRIC REDOX TITRATIONS ($K_2Cr_2O_7$ VS FAS)

CALCULATION

Volume of $K_2Cr_2O_7$ (V_1)	= V_1 ml
Normality of $K_2Cr_2O_7$ (V_1)	= y N
Volume of FAS (V_2)	= 20 ml
Normality of FAS (N_2)	= (V_1 x y/20) N

8B. POTENTIOMETRIC REDOX TITRATIONS ($K_2Cr_2O_7$ VS FAS)

PROCEDURE :

20 ml of FAS was pipetted out into a 250ml beaker and an equal volume of 2N sulphuric acid is added to it. A clean platinum electrode was introduced into the solution. It was connected to a calomel electrode through KCl salt bridge. The two electrodes were connected to a digital voltmeter to get a positive emf value. $K_2Cr_2O_7$ solution was taken in a clean burette. The burette was so maintained above the beakers that its tip was just above the liquid. The trial experiment was done by adding 1 ml portion at a time noting the emf value for each addition. This trial titration gave the equivalence point within 1 ml range. Now another 20 ml of FAS solution was pipetted out into a clean beaker added an equal volume of 2N H_2SO_4following the electrodes were introduced as before. $K_2Cr_2O_7$ solution was added from the burette in bulk till its volume 1 ml short of the range fixed in the previous trial experiment. The solution was stirred well and the emf of the cell was measured. Now 0.1 ml portion of the titrant was added at a time and for each addition emfwas noted.

A graph was plotted connecting volume of $K_2Cr_2O_7$ added and emf. The accurate end point was determined by plotting E/V Vs volume of $K_2Cr_2O_7$ added. From the end point strength of FAS solution was calculated.

RESULT :

Normality of FAS = N

INFERENCE:

Rise in EMF is high nearer to the equivalence point and then begins to decrease

NO. OF BENEFICIARIES:43

OUTCOME:

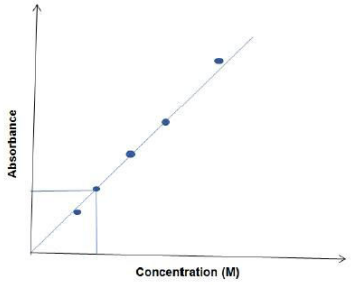
Students are able to construct the electrochemical cell, operate the Potentiometer and to determine the normality of the given FAS potentiometrically.

9. VERIFICATION OF BEER-LAMBERT'S LAW BY SPECTROPHOTOMETRY

λ_{max}=526.5 nm

Concentration of $KMnO_4$ (M)	Absorbance
0.002	
0.004	
0.006	
0.008	
0.01	
given	

CALIBRATION GRAPH



9. VERIFICATION OF BEER-LAMBERT'S LAW BY SPECTROPHOTOMETRY

AIM

To verify Beer-Lambert's law and to determine the concentration of given potassium permanganate solution by spectrometric method.

REQUIREMENTS:

EQUIPMENTS	:Weighing balance,spectrophotometer,
CHEMICALS	:potassium permanganate
GLASSWARES	:Weighing bottle, pipette – 20ml, 10ml, standard measuring flasks –50ml

PRINCIPLE:

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

$$A= \log(I_0/I_t)=\varepsilon cl$$

where,

$\log(I_0/I_t)$is the optical density at λ_{max}

ε is the molar extinction of co-efficient

c is the concentration of the sample

l is the path length of the sample in cm

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

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

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

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

Before verification of Beer's law, it is necessary to select a suitable wave length and determines whether Beer's law is valid at the wavelength selected. The most suitable

wavelength is that at which maximum absorbance is observed, called λ_{max}. The λ_{max} will always be at the same wavelength for a given solution under any condition.

PROCEDURE

A stock solution of 0.01M potassium permanganate solution was prepared. The uv - visible spectrum of the solution was recorded in a Shimadzu UV spectrometer. The spectra should characteristic absorption band in the visible region. The peak at 526.5 nm was chosen for the measurement of optical density.

From the stock solution of potassium permanganate solution (0.01M)a series of solution with known concentration(0.002M, 0.004M, 0.006M, 0.008M)were prepared by dilution. The optical density of these solutions are measured at 526.5 nm. A calibration curve was drawn by plotting optical density (y-axis) against concentration (x-axis).A straight line passing through origin was obtained. Molar extinction coefficient can be determined from the slope of the line. The given $KMnO_4$ solution was made up to the mark in a 100 ml standard measuring flask and the optical density was measured and the Strength of given potassium permanganate solution was noted from the graph.

RESULT:

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

Concentration of given potassium permanganate solution = M

INFERENCE:

As Concentration increases, absorbance increases. Hence the straight line plot is obtained.

NO. OF BENEFICIARIES:43

OUTCOME:

Students are able to experimentally verify the Beer-Lamberts law and to determine the concentration of the given solution spectrophotometrically from the calibration curve.