

Scope

The course includes practical exercises that illustrate some of the electrochemical techniques, and it gives a survey of electrochemical instrumentation.

Objectives

It enables the students to apply their knowledge in laboratory experiments like

1. Determination of critical solution temperature and eutectic temperature
2. To determine distribution coefficients
3. To carry out the titrations potentiometrically.

Methodology

Potentiometer, electrochemical experiments

Phase Equilibria:

- I. Determination of critical solution temperature and composition at CST of the phenol-water system and to study the effect of impurities of sodium chloride and succinic acid on it.
- II. Phase equilibria: Construction of the phase diagram using cooling curves or ignition tube method: a. simple eutectic and b. congruently melting systems.
- III. Distribution of acetic/ benzoic acid between water and chloroform or cyclohexane.
- IV. Study the equilibrium of at least one of the following reactions by the distribution method:
 - (i) $I_2(aq) + I^-(aq) \rightarrow I_3^-(aq)$
 - (ii) $Cu^{2+}(aq) + nNH_3 \rightarrow Cu(NH_3)_n^{2+}$

Potentiometry:

- V. Perform the following potentiometric titrations: i. Strong acid vs. Strong base ii. Weak acid vs. Strong base iii. Dibasic acid vs. Strong base iv. Potassium dichromate vs. Mohr's salt

Suggested Readings

Text Books:

1. Khosla, B. D., Garg, V. C. & Gulati, A. (2011). *Senior Practical Physical Chemistry*. 25. New Delhi: R. Chand & Co.
2. Garland, C. W.; Nibler, J. W. & Shoemaker, D. P. (2003). *Experiments in Physical Chemistry*. 8th Ed. McGraw-Hill: New York.

Reference Books

1. Halpern, A. M. & McBane, G. C. (2003). *Experimental Physical Chemistry*. 3rd Ed. New York: W. H. Freeman & Co.

List of Experiments



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

List of Experiments

Name of the Staff : **R Kumar**
Department : **Chemistry**
Subject : **Phase Equilibria and Chemical Kinetics-Practical**
Subject Code : **18CHU311**
Class : **II B.Sc Chemistry**
Year and Semester : **II / III**

S.No	NAME OF THE EXPERIMENT
1	Phase Diagram-Simple Eutectic System
2	Determination of CST of Phenol-Water System
3	Effect of impurity on CST of Phenol-Succinic acid system
4	Determination of CST of Phenol-NaCl System
5	Potentiometric titration- Acid-Base titration
6	Redox titration -KMnO ₄ Vs Fe (II)
7	Determination of partition coefficient of benzoic acid between Benzene and Water.
8	Determination of equilibrium constant for the reaction between Potassium iodide and iodine.

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Course Name:Phase equilibria and chemical kinetics
practicals

Course Code:18CHU311

Batch:2018-2021

**PHASE EQUILIBRIA AND CHEMICAL KINETICS PRACTICAL
LAB MANUAL
FOR**

II B.Sc., CHEMISTRY STUDENTS



**DEPARTMENT OF CHEMISTRY
KARPAGAM ACADEMY OF HIGHER EDUCATION**

(Deemed to be University established Under section 3 of UGC Act, 1956)

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4	Determination of partition coefficient of benzoic acid between Benzene and Water.	14
5	Determination of equilibrium constant for the reaction between Potassium iodide and iodine.	16

Experiment No 1**DETERMINATION OF CRITICAL SOLUTION TEMPERATURE PHENOL – WATER SYSTEM****Aim**

To determine the critical solution temperature phenol – water system.

Principle

The temperature at which two partially miscible liquids become completely miscible is called critical solution temperature. mixtures of phenol and water of varying compositions are taken and their miscibility temperatures are plotted against their compositions. the maximum temperature point on the curve obtained is the critical solution temperature.

Materials Required

Flat bottom test tube, thermometer, burette, water bath, phenol,

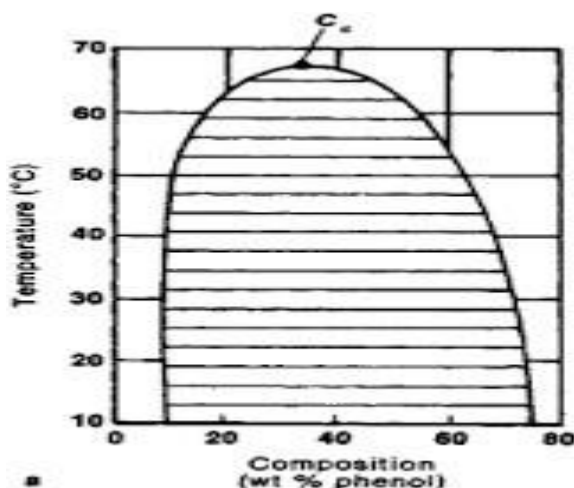
Procedure

An accurately weighted amount of phenol (about 2g) is taken in a flat bottomed test tube. it is fitted with a cork carrying a thermometer and a stirrer. 2ml of water is added to it from burette. the test tube is fixed in a bigger tube by a cork and the whole is heated at which the two liquids become completely miscible is noted. the mixture is then cooled by cooling the water bath, and the temperature at which the turbidity appears is noted. the average value is taken as the miscibility temperature.

The experiment is repeated by adding 2, 4, 6, 8, 10, 12, 14, 16ml of water and their corresponding miscibility temperature are noted. the results are tabulated. the results obtained by such an experiment are given below. These miscibility temperatures are plotted against their respective compositions. the maximum temperatures on the curve is taken as the critical solution temperature. according to the above data the critical solution temperature of phenol – water system is found to be 68.5°C.

Observations and calculations

Expt.	mass of phenol in g	mass of water in gram	percentage of phenol	miscibility temperature in degree centigrade

Model graph**Results**

The critical solution temperature of phenol water system is =

Note

Note in the case of certain systems the mutual solubility of the two components decreases with the rise of temperature. This shows that they have a lower critical solution temperature. The critical solution temperature is markedly influenced by the presence of impurities.

Viva questions

1. What is critical solution temperature?
2. Define miscibility?
3. Define mutual solubility of the solution?

Experiment 2.**Determination of critical solution temperature and composition at CST of the phenol water system and to study the effect of impurities of sodium chloride****Aim**

To determine the Critical Solution temperature (CST) for the phenol-water system, and to study the effect of added impurity (NaCl) on the CST.

Principle

The number of homogeneous, mechanically separable and physically distinct parts of a heterogeneous system is known as the number of phases, **P**, of the system.

Each phase is separated from other phases by a physical boundary.

When an equilibrium exists between a number of phases under external controlling conditions such as temperature, pressure, and concentration, the following relationship holds good :

$$P + F = C + 2 \quad \text{-->} \quad (1)$$

where **P = number of Phases in equilibrium,**
C = number of Components in the system,
and F = number of degrees of Freedom.

Equation (1) is called the **Phase Rule**, which relates the phases, components and degrees of freedom of the system. The different terms used in Eq. (1) may be defined as follows :

Phase is defined as any homogeneous and physically distinct part of a system which is separated from other parts of the system by definite bounding surfaces.

The number of **Components** in a system in equilibrium is the smallest number of independently variable constituents by means of which the composition of each phase present can be expressed either directly, or in the form of a chemical equation.

The number of **degrees of freedom** or **variance** is the number of variable factors, such as temperature, pressure and concentration, which need to be fixed in order to completely define the conditions of a system in equilibrium.

When 2 partially miscible liquids are mixed and shaken together, we get 2 solutions of different compositions. For example, on shaking phenol and water, we get 2 layers : **the upper layer** is a

solution of **water in phenol**, and the **lower layer** is a solution of **phenol in water**. At a fixed temperature, the composition of each solution is fixed, and both the solutions are in equilibrium.

Two solutions of different compositions existing in equilibrium with one another are known as **conjugate solutions**. **Above a particular temperature, such solutions are completely miscible in all proportions. Such a temperature is known as the Critical Solution Temperature (CST) or Consolute Temperature.** As the mutual solubility increases with temperature in this particular case, it is known as **Upper Consolute Temperature**.

Mutual Solubility Curve

If we have 2 liquids **A** and **B** and we mix them, we get a mixture of composition **c1**. At any temperature **t1^oC** (or below t1^oC), the 2 liquids separate into 2 layers of different compositions. Above t1^oC, the 2 layers are completely miscible. Thus, the point corresponding to temperature t1^oC and composition c1 is known as the **miscibility point**.

If we take another mixture of **A** and **B** of composition c2, we can find out the temperature (say **t2^oC**) above which the last 2 layers become completely miscible. Similarly, we can find out corresponding temperatures for a number of mixtures of **A** and **B**.

If a curve is plotted with **temperature (°C)** as ordinate (**y-axis**) against **concentration (% by weight)** as abscissa (**x-axis**), we get a **mutual solubility curve**

It is found that above B, the 2 liquids will become miscible in all proportions, and hence it is known as the

Critical Solution Temperature (CST).

Above this temperature, the liquid mixture is homogeneous.

Below this temperature, the mixture separates into 2 layers.

The **CST** is markedly affected by pressure and also by the presence of impurities. Hence, the **CST** may be taken as a criterion for the purity of a substance.

The solubility changes in the CST for Phenol - Water system is affected by adding an electrolyte to it on miscibility temperature.

If a graph is plotted with mean miscible temperature as ordinate (y-axis) against concentration of electrolyte (N) as ordinate (x-axis), we get the unknown concentration of electrolyte. From that, we can estimate the amount of solution supplied.

Materials Required

Hard Glass Test tubes (or boiling tube as air jacket), thermometer (graduated to 0.1^oC), stirrer, beakers (500ml, 100 ml), volumetric flask (100 ml), pipette (1 ml, 2 ml), phenol, distilled water, sodium chloride, hot plate.

Procedure**PART – A: CST for Phenol - Water system**

1. Weigh out about 5.0 g of phenol in a dry boiling tube.
2. Add **2.0 ml of distilled water**. The solution is stirred.
3. Heat the solution in a water bath, with continuous stirring. <http://start.fedoraproject.org/>
4. At a certain temperature, the mixture becomes **clear**. Note this temperature (**t₁^oC**).
5. Remove the tube from the water bath, and allow the solution to cool down slowly. Note the temperature at which the **turbidity re-appears** (**t₂^oC**).
6. Repeat Steps 2 to 6, after each addition of 2 ml of solution, followed by heating and subsequent cooling,

Note

The temperature of disappearance of turbidity, and the temperature of the re-appearance of turbidity. The observation is that the temperature (^oC) of complete miscibility rises, reaches a maximum value, and then decreases.

PART – B: CST for Phenol – NaCl system

1. Weigh out about 5.0 g of phenol in a dry boiling tube.
2. Add **2.0 ml of 0.1 N NaCl** solution. The solution is stirred.
3. Repeat the steps 3 to 8 and continue the experiment by following the part – A.

PART – C: Effect of Impurity (NaCl) on CST

1. Following proportions of solutions are to be prepared given in the table 2.
2. The clearing and clouding temperature for the prepared solutions are noted and their mean value is taken.
3. From the mean miscibility temperature of unknown solution, the concentration (N) corresponding to them are obtained from the given values of NaCl solution and the volume of electrolyte solution supplied is calculated.

Observations

Room Temperature =^oC

$$\% \text{ of Phenol} = \frac{(\text{Volume of phenol} \times \text{Density of phenol})}{(\text{Volume of phenol} \times \text{Density of phenol}) + (\text{Volume of H}_2\text{O or NaCl})}$$

Weight of Phenol =

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Density of Phenol = _____

_____ g/m

l Density of water (d) =

_____ g/m

l

Density of NaCl Solution (0.1N) = _____ g/ml

5.	5.0	x1	y1				
6.	5.0	x2	y2				

Calculations

1. A curve is plotted with **miscibility temperature ($^{\circ}\text{C}$)** as ordinate against **concentration of phenol (percentage by weight)** as abscissa.
2. This curve will be the **mutual solubility curve** of the **phenol – water system**.
3. The maximum of the solubility curve will give the value of the **Critical Solution Temperature**.
4. A graph is plotted with **mean miscibility temperature ($^{\circ}$)** as **ordinate** against **concentration of NaCl (N)** as **abscissa**.
5. This graph gives the unknown concentration of electrolyte solution supplied.

NOTE :

- (i) The temperature of the solution should be increased or decreased **very slowly**.
 - (ii) The mixture of phenol and water should be **continuously and uniformly stirred**.
 - (iii) The stirrer should not touch the bottom of the boiling tube.
- Care should be taken while handling phenol.

Result

- 1.(i) **The Critical Solution Temperature for the Phenol – Water system-----**
 (ii) **% of phenol at CST-----**
- 2.(i) **In the presence of NaCl, the CST for the Phenol - Water system-----**
 (ii) **% of phenol at CST-----**

Experiment No 3**PHASE DIAGRAM OF PARA –NITROTOLUENE AND NAPHTHALENE****Aim**

Phase diagram of Para –nitrotoluene and naphthalene

Principle

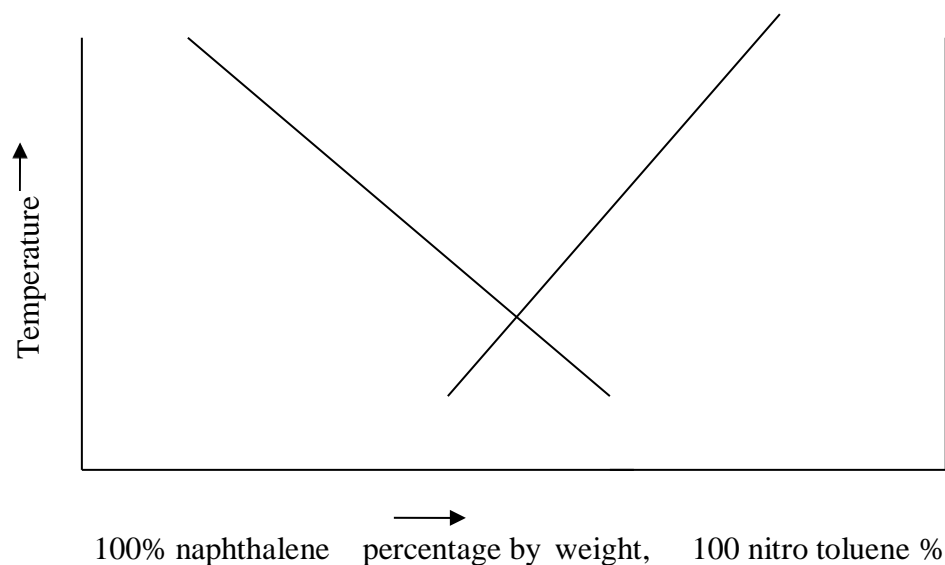
Para-nitro toluene and naphthalene form a two component system of the simple eutectic type. Mixtures of the two substances of varying compositions are taken and the freezing points of each melted solid solution are determined. The freezing point of a series of liquid mixtures of varying compositions from one pure component to other are determined and plotted against the corresponding composition of the liquid when two curves are obtained which will meet a minimum point. This is the lowest temperature at which the liquid mixtures of the two components will freeze and hence is called the eutectic point.

Materials Required

Para-nitro toluene, naphthalene, thermometer.

Procedure

A hard glass tube is cleaned and dried. A known accurate mass of para-nitro toluene (about 15g) is weighed into it. An accurately weighed amount of naphthalene is also transferred into it so that it would constitute about 20% of the mixture. This tube is fitted onto an air jacket immersed in water bath which is heated over a burner. A sensitive thermometer and a clean stirrer is provided for the tube containing the mixture. The heating is continued until the mixture is melted and the temperature is about 10°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. Record the temperature reading every half minute while stirring the liquid constantly until the arrest in cooling is reached, the temperature of arrest will be the freezing point of the pure component. Now a further weighed amount of naphthalene is added so that it would constitute about 30%. Then the freezing point is determined as described for naphthalene. The experiment is repeated for 40, 50, 60 and 70% percentage of naphthalene. The freezing points of the pure components are also found out. The result may be tabulated. Results obtained in such an experiment are given below.

**Observations and calculation**

Expt no.	Mass of p-nitro toluene in g	Mass of naphthalene in g	Percentage of naphthalene	Freezing point

A graph is drawn with composition on x-axis and melting points on the y-axis. The resulting graph has a minimum temperature point which is eutectic point. The point is actually got by drawing smooth, free hand curves through the plotted points on either side of the eutectic and finding the point where they meet. The eutectic composition is read from the graph.

Results

The eutectic composition of Para nitro toluene and naphthalene system =

Note

- To get accurate values, the loss of the heat due to radiation and other should be minimized as far as possible.

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- i. Super heating and super cooling are to be avoided.
- ii. The temperature should be noted after mixing the components thoroughly. It is noted after the whole mixture has melted and when the first crystals appear on cooling.
- iv. A number of reading should be taken get more concurrent values and the average of the readings are plotted.

Viva questions

- 1. Define phase diagram..?
- 2. Write the structure for Para nitro toluene and naphthalene .?
- 3. How the phase diagram of Para nitro toluene and naphthalene differ from naphthalene – biphenyl amine system.?
- 4. Define two component system of the eutectic type.?
- 5. Define eutectic composition.?
- 6. What do you meant by super heating and super cooling.?

Experiment No.4**Association Factor of Benzoic Acid between Benzene and Water**

To determine the association factor of benzoic acid between Benzene and water.

Principle

When a solute like benzoic acid is added to a mixture of two immiscible liquids, solute distributes itself between the liquids. The ratio of the concentration of benzoic acid in the two layers is constant. The ratio of the concentrations is called partition or distribution coefficient. It is independent of the amount of the substance added to the liquid pair.

$$K = \frac{C_1}{\sqrt{C_2}}$$

$$K = \frac{[\text{Benzoic acid}]_{\text{aqueous layer}}}{\sqrt{[\text{Benzoic acid}]_{\text{organic layer}}}}$$

Taking logarithms and simplifying the equations

$$n = \frac{\log C_2 - \log C_2'}{\log C_1 - \log C_1'}$$

n = association factor for benzoic acid C_1 and C_2 terms refer to the concentration of benzene in aqueous and organic layers.

Materials required

- (i) Benzene
- (ii) Distilled water
- (iii) Benzoic acid
- (iv) N/10 NaOH
- (v) N/100 NaOH
- (vi) Phenolphthalein indicator
- (vii) Burette -50ml-2 Nos
- (viii) Pipette 10 ml
- (ix) Reaction bottle 250 ml – 2 Nos

Procedure:

In a clean, dry reaction bottle 50 ml of benzene and exactly 50 ml of distilled water are added followed by 2 g of benzoic acid. In another reaction bottle, 40 ml of benzene, 60 ml distilled water and 2 g of benzoic acid are added. The contents of the reaction bottle are thoroughly shaken well nearly for an hour and set aside in a trough containing water to reach and equilibrium stage. In a reaction bottle the organic layer (upper layer) and the aqueous layer (lower layer) are distinctly separated. Exactly 10 ml of the organic layer is pipetted out into a clean conical flask, and a drop of phenolphthalein indicator is added. It is titrated against standard N/10 NaOH solution until a pale pink colour appears. The titration is repeated for concordant value. In a similar way 10 ml of the aqueous layer is carefully pipetted out and titrated against N/100 NaOH solution taken in the burette using phenolphthalein indicator. From the titre values the

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concentration of benzoic acid in each layer and hence its association factor in one of the solvents is calculated.

Observations and calculation

S.No	Type of layer	Volume pipette out	Burette reading		Volume of N/10 NaOH	Normality N/100 NaOH
			Initial	Final		
Bottle I 50ml benzene + 50 ml of water + 2 g benzoic acid	Organic layer	10 ml	0			
		10 ml	0			
	Aqueous layer	10 ml	0			
		10 ml	0			
Bottle II 40 ml benzene + 60 ml of water + 2 g of benzoic acid	Organic layer	10 ml	0			
		10 ml	0			
	Aqueous layer	10 ml	0			
		10 ml	0			

Calculations:

$$K = \frac{C_1}{\sqrt{C_2}}$$

$$K = \frac{[\text{Benzoic acid}]_{\text{aqueous layer}}}{\sqrt{[\text{Benzoic acid}]_{\text{organic layer}}}}$$

Taking logarithms and simplifying the equations

$$n = \frac{\log C_2 - \log C_2'}{\log C_1 - \log C_1'}$$

n = association factor for benzoic acid C_1 and C_2 terms refer to the concentration of benzene in aqueous and organic layers.

Result:

The association factor for benzoic acid between benzene and water n=-----

Viva questions

1. Define Nernst distribution law.?
2. Define distribution co-efficient.?

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

Experiment No.5**DETERMINATION OF EQUILIBRIUM CONSTANT BY DISTRIBUTION METHOD****Aim**

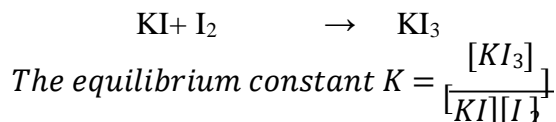
To determination of equilibrium constant of the reaction between potassium iodine and iodine to find out the concentration of the given potassium iodide solution by distribution method

Principle

The partition law is obeyed Well for the distribution of iodine between CCl_4 and water. At constant temperature the partition co-efficient is given by

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

The law doesn't hold good in the distribution of iodine between organic and aqueous layer of KI because in aqueous layer the complex is formed. The failure of distribution law is only apparent and it is expected to hold good provided the concentration of iodine in aqueous layer is taken into a consideration of iodine is distributed between CCl_4 and aqueous solution of KI, the total concentration of iodine in aqueous layer is given by [concentration of iodine in free + concentration of I_3^-] which can be determined by titration with standard thio solution. The concentration of iodine in organic layer can also be determined by titration with standard thio solution. Then the concentration of free iodine in aqueous layer can be determined by dividing the concentration of iodine in organic layer by partition co-efficient. It is used to calculate the values of Cl_3^- and Cl_2 . If the initial concentration of KI is known and its Cl_3^- , Cl_2 , Cl^- are unknown the equilibrium constant for the reaction.



The equilibrium constant can be calculated by knowing the concentration of free iodine and potassium iodide. If the equilibrium constant is known, the concentration of the unknown solution can be calculated.

Materials required

- (i) Iodine in CCl_4 solution
- (ii) M/10 KI solution
- (iii) N/50 Thio solution
- (iv) Starch indicator solution
- (v) Burette
- (vi) Pipette 10 ml

- (vii) Conical flask
(viii) Reaction bottle 250 ml – 2 Nos

Procedure

Exactly 50 ml of M/10 KI solution taken in a clean stoppered reaction bottle is mixed with exactly benzene and exactly 50 ml of a standard solution of iodine in carbon tetrachloride. The mixture is mechanically shaken well for about an hour. If mechanical shaking machine is not available, the bottle containing the mixture must be thoroughly shaken nearly an hour. It is then set aside for about 20 minutes in a water trough so that equilibrium is attained. Exactly 5 ml of the aqueous layer is pipette out into a clean conical flask. A little of KI salt is added to it. The liberated iodine is titrated against standard sodium thiosulphate solution taken in a clean burette, using starch as indicator. The end point is the disappearance of blue colour. Another titration is performed to determine the concordant values. From the titre value the concentration of iodine in aqueous layer is calculated. In a similar manner, the concentration of iodine in organic layer is calculated. Using these values, the equilibrium constant is calculated.

The given KI solution is made up to 100 ml in a standard flask. After thorough shaking to make the solution uniform, exactly 50 ml of the solution is taken in a reaction bottle. It is then mixed with exactly 50 ml of iodine in CCl_4 solution. The bottle is shaken well for about an hour and set aside for about 20 minutes to attain the equilibrium. The concentration of iodine in aqueous layer is found out as described above. From the concentration of iodine and knowing the equilibrium constant, the concentration of given KI solution is calculated.

Observations and calculations

	Type of layer	Volume pipette out	Burette reading		Volume of thio	Normality of thio
			Initial	Final		
Bottle I 50 ml of I_2 in CCl_4 + 50 ml of M/10 KI solution	Organic layer	10 ml 10 ml	0 0			
	Aqueous layer	10 ml 10 ml	0 0			
Bottle II 50 ml of I_2 in CCl_4 + 50 ml of M/10 KI solution	Organic layer	10 ml 10 ml	0 0			
	Aqueous layer	10 ml 10 ml	0 0			

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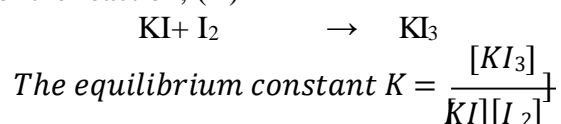
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The equilibrium constant of the reaction, (K)



Where,

 $[\text{KI}_3]$ = Concentration of I_3^- at equilibrium $[\text{KI}]$ = Concentration of KI at equilibrium or unreacted $[\text{I}_2]$ = concentration of free iodine in aqueous layer,

Suppose the concentration of free I_2 in organic layer is C_1 moles /lit and in aqueous layer is C_2 moles / lit. Let the initial concentration of KI be C_3 moles / lit. The partition coefficient of iodine between CCl_4 and water is 80. ($K_D = 80$)

Bottle I organic layer:

Volume of sodium thio sulphate (v_1) =
 Normality of sodium thio sulphate (N_1) = Volume
 of organic layer pipetted out (V_2) = Normality of
 iodine in organic layer (N_2) $= v_1 \times \frac{N_1}{N_2}$

Concentration of iodine in organic layer c_1 =**Aqueous layer:**

Volume of sodium thio sulphate (v_1) =
 Normality of sodium thio sulphate (N_1) =
 Volume of organic layer pipetted out (V_2) =
 Normality of iodine in organic layer (N_2) $= v_1 \times \frac{N_1}{N_2}$

Concentration of iodine in aqueous layer c_2 =Concentration of iodine in aqueous layer c_3 = $\frac{c_1}{K_D}$ Concentration of KI_3 ($c_2 - C_3$) = C_4 --- gmmol/lit

Concentration of KI used 0.1 mol/lit

Concentration of KI at equilibrium = $0.1 - c_4$ ----- gmmol/lit

Equilibrium constant $\frac{[\text{KI}_3]}{[\text{KI}][\text{I}_2]} =$

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Result

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

- (i)
- (ii)