

**B.Sc. Programme
(Chemistry Major)**

Laboratory Manual

Physical Chemistry Experiments



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TRANSITION

DETERMINATION OF TRANSITION TEMPERATURE

AIM

To determine the transition temperature of the given substance.

PROCEDURE :

The transition temperature is the temperature at which the two crystalline forms are at equilibrium. The temperature remains constant until one crystalline form is completely converted into another form.

About 4g of the crystalline substance was taken in a specimen tube and 4ml of liquid paraffin was added into it. The specimen tube is fitted with outer jacket provided with a stirrer and sensitive thermometer. The specimen tube was heated in a water bath using very small flame with constant stirring. The temperature was noted for every half a minute. At a particular temperature, it remains constant and even after this, the bath was heated to 2°C above and this temperature was also noted for every half a minute, while cooling also time and temperature were noted.

The temperature versus time graph was drawn for both heating and cooling. The transition temperature was noted from the graph.

Result :

The transition temperature of the given salt hydrate =

PHENOL IN WATER

S. No.	Volume of Water in ml	Volume of Phenol in ml	Weight percentage of phenol (%)	Miscibility temperature (°C)		
				Heating	Cooling	Average
1						
2						
3						
4						
5						

WATER IN PHENOL

S. No.	Volume of Phenol in ml	Volume of Water in ml	Weight percentage of phenol (%)	Miscibility temperature (°C)		
				Heating	Cooling	Average
1						
2						
3						
4						
5						

PHENOL WATER SYSTEM – I

DETERMINATION OF CONSOLUTE TEMPERATURE

AIM :

To determine the consolute temperature of phenol water system and the composition of phenol at consolute temperature.

PRINCIPLE

In the case of partially miscible system solubility increases with temperature. Hence, it may be anticipated that liquid which are partially miscible at room temperature are completely miscible at high temperature. In the case of phenol water system, there is a separation into two layers upto certain temperature. But above that temperature phenol and water are completely miscible in all proportions. This temperature is known as the consolute temperature of phenol water system.

PROCEDURE

2.5ml of phenol was taken in a dry specimen tube provided with a stirrer and an outer jacket. 0.5ml of water was added with this and suspended in a water bath which is gradually heated. The temperature at which the turbidity just disappears was noted. The specimen tube was taken out and cooled with constant stirring. The temperature at which the turbidity reappears was noted. The average of these two temperatures was calculated. The experiment was repeated for four more additions of water in 0.5ml proportion. Similarly 2.5ml of water was taken in a specimen tube and phenol was added for four more times in 0.5ml proportion. After each addition, the temperature at which the turbidity disappears on heating and reappears on cooling were noted.

A graph was drawn by plotting miscibility temperature on Y-axis and percentage composition of phenol on X-axis. The temperature corresponding to maximum above which phenol and water were completely miscible in all proportions gives the consolute temperature and the composition corresponding to that temperature gives the percentage composition of phenol.

Result :

- (i) The consolute temperature of phenol water system =
- (ii) The composition of phenol at consolute temperature =

WATER IN PHENOL

S. No.	Volume of Phenol in ml	Volume of Sodium chloride in ml	Percentage of Sodium chloride (%)	Miscibility temperature (°C)		
				Heating	Cooling	Average
1						
2						
3						
4						
5						
6						
7						

PHENOL WATER SYSTEM – II
EFFECT OF ELECTROLYTE ON CONSOLUTE TEMPERATURE

AIM :

To study the influence of third substance on consolute temperature of phenol water system and to estimate the amount of sodium chloride in a given system.

PRINCIPLE :

The consolute temperature of any system is greatly affected by the presence of soluble substance in the system. The consolute temperature of phenol water system is raised by the addition of sodium chloride.

PROCEDURE :

Exactly 2% of sodium chloride was prepared and diluted to 0.5%, 0.4%, 0.3%, 0.2% and 0.1%. 2.5ml of phenol was taken in a specimen tube and 2.5ml of 0.1% of sodium chloride was added to it and temperature of complete miscibility was found out. The same procedure was repeated for all other solutions of sodium chloride of different concentration.

The given solution was made upto 50ml and 2.5ml of this solution is mixed with 2.5ml of phenol and miscibility point was found out. A graph was drawn by plotting miscibility temperature on Y-axis and percentage of NaCl on X-axis. The concentration of the unknown sodium chloride solution was found out by extrapolation.

RESULT :

The concentration of given sodium chloride solutions

Unknown A =

Unknown B =

Freezing point of a solvent	
Time in Minutes	Temp. in °C

Freezing point of the solution	
Time in Minutes	Temp. in °C

CALCULATION :

Weight of the solvent (W_1) =

Weight of the solute (W_2) =

Freezing point of the solvent (T_1) =

Freezing point of the solution (T_2) =

Depression in freezing point (ΔT_f) = ($T_1 - T_2$)

=

=

Molecular weight of the solvent (M_2) =

Molal depression constant of the solvent (K_f) = $K_f = \frac{\Delta T_f \times W_1 \times M_2}{1000 \times W_2}$

=

=

=

RAST'S MACRO METHOD – I
DETERMINATION OF MOLAL DEPRESSION CONSTANT

AIM :

To determine the molal depression constant of the given substance.

PROCEDURE :

Weighed quantity of the given solvent (about 4g) was taken in a specimen tube surrounded by an outer 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. About 0.4g of the given solute was added to the molten solvent and heated. When the molten mixture of the liquid become homogeneous, it was allowed to freeze with constant stirring and the freezing point was noted. The difference between the freezing point of the solvent and that of the mixture gives the depression in freezing point. The molal depression constant calculated using the formula.

$$K_f = \frac{\Delta T_f \times W_1 \times M_2}{1000 \times W_2}$$

where,

ΔT_f	=	Depression in freezing point
W_1	=	Weight of the solvent
W_2	=	Weight of the solute
M_2	=	Molecular weight of the solute
K_f	=	Molal depression constant

RESULT :

Molal depression constant of the given solvent =

Freezing point of a solvent	
Time in Minutes	Temp. in °C

Freezing point of the solution	
Time in Minutes	Temp. in °C

CALCULATION :

Weight of the solvent (W_1) =

Weight of the solute (W_2) =

Freezing point of the solvent (T_1) =

Freezing point of the solution (T_2) =

Depression in freezing point (ΔT_f) =

Molal depression constant of the solvent (K_f) =

Molecular weight of the solute (M_2) = $\frac{K_f \times 1000 \times W_2}{\Delta T_f \times W_1}$

=

=

M_2 =

RAST'S MACRO METHOD – II
DETERMINATION OF MOLECULAR WEIGHT

AIM :

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

PROCEDURE :

Weighed quantity of the given solvent (about 4g) was taken in a specimen tube surrounded by an outer jacket. This was kept in a water bath and allowed to melt completely. The molten liquid was allowed to cool with constant stirring. The freezing point was noted. Then weighed about 0.4g of the unknown solute accurately and it was added to the solvent. The mixture was melted and when the molten mixture become homogeneous it was allowed to freeze with constant stirring and noted the freezing point of the mixture. The difference between the freezing point of the solvent and that of the mixture gives depression in freezing point.

Knowing the molal depression constant, the molecular weight of the given substance was calculated using the formula.

$$M_2 = \frac{K_f \times 1000 \times W_2}{\Delta T_f \times W_1}$$

RESULT :

The molecular weight of the given unknown solute =

S. No.	Time in minutes	Burette Reading (ml)		Volume of NaOH (ml)	$V_{\infty} - V_t$ ml	log ($V_{\infty} - V_t$)	Rate constant $\times 10^{-3} \text{ min}^{-1}$
		Initial	Final				
1							
2							
3							
4							
5							
6							
7							
8							
				Average (k) = $\quad \quad \quad \times 10^{-3} \text{ min}^{-1}$			

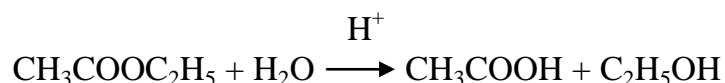
KINETICS HYDROLYSIS OF AN ESTER

AIM :

To determine the rate constant for the hydrolysis of an ester with an acid as a catalyst.

PRINCIPLE :

When an ester such as ethyl acetate reacts with water it is converted into an acid and an alcohol. The hydrolysis takes place very slowly and is catalysed by an acid and the velocity depends upon the strength of an acid. The reaction is pseudounimolecular, when water is present in excess.



PROCEDURE :

100ml of N/2 hydrochloric acid is buretted out in a dry conical flask and kept into a water bath at room temperature. The ester is kept in the same temperature for half an hour in a boiling test tube. After half an hour, 10ml of ester is pipetted out into a conical flask containing acid. A stop clock is started, when half of the ester is added. The contents are shaken 5ml of the mixture is withdrawn and poured into clean conical flask containing ice pieces and a drop of phenolphthalein. The acid liberated is titrated against approximately N/5 NaOH solution. The titrations are repeated at regular intervals of 10 minutes upto 60 minutes. The remaining solution is heated for an hour in a water bath at about (55–60°C) to allow the reaction to complete. This is then cooled at room temperature and 5ml of solution is withdrawn and added a drop of phenolphthalein and titrated against same NaOH. The velocity constant is calculated using the formula.

$$K = \frac{2.303}{t} \log \left[\frac{V_\infty - V_0}{V_\infty - V_t} \right] \text{min}^{-1}$$

where,

t	=	Time in minutes
V ₀	=	Volume of NaOH in ml
V _t	=	Volume of NaOH in ml at corresponding time.
V _∞	=	Volume of NaOH in ml at ∞ time.

RESULT :

The rate constant for hydrolysis of an ester using the given acid as catalyst

is $k_{\text{cal}} =$

$k_{\text{graph}} =$

Volume of NaOH (ml)	Conductance in milli.mho	Volume of NaOH (ml)	Conductance in milli.mho

Calculation :

Determination of Strength of NaOH

Volume of hydrochloric acid (V_1) =

Strength of hydrochloric acid (N_1) =

Volume of Sodium hydroxide (V_2) =

Strength of Sodium hydroxide (N_2) =

$$V_1 N_1 = V_2 N_2$$

$$N_2 = \frac{V_1 N_1}{V_2}$$

=

Strength of Sodium hydroxide = N

CONDUCTOMETRY

ACID – BASE TITRATION

AIM :

To determine the concentration of Sodium hydroxide using standard hydrochloric acid by conductometric titration.

PROCEDURE :

40ml of hydrochloric acid (0.05N) was pipetted out into a 100ml beaker and the conductivity cell was placed in it. Then water was added so that the electrodes were well immersed in the solution. Sodium hydroxide of strength approximately 0.25N was added in 0.5ml proportions from the burette to the beaker containing HCl. After each addition the solution was stirred and allowed to attain equilibrium. The conductance was noted. The conductance was plotted against the volume of sodium hydroxide solution added. The end point could be obtained from the point of intersection.

RESULT :

The strength of the given sodium hydroxide solution = N

S. No.	Varying concentration of KCl in N	Specific conductance (k) $\text{ohm}^{-1}\text{cm}^{-1} \times 10^{-3}$	Equivalent conductance $\left(K \times \frac{1000}{C} \right)$ $\text{ohm}^{-1}\text{cm}^2 \text{g.equ}^{-1}$
1			
2			
3			
4			
5			
6			
7			
8			

Calculation :

(i) $\lambda_c =$ =

=

(ii) $\lambda_c =$ =

=

(iii) $\lambda_c =$ =

=

(iv) $\lambda_c =$ =

=

(v) $\lambda_c =$ =

=

(vi) $\lambda_c =$ =

=

(vii) $\lambda_c =$ =

=

(viii) $\lambda_c =$ =

=

CONDUCTOMETRY

DETERMINATION OF EQUIVALENT CONDUCTANCE

AIM :

To determine the equivalent conductance of the given electrolyte solution and to study the effect of dilution on equivalent conductance.

PROCEDURE :

Prepared 250ml of 0.1N (exact) solution of KCl by weighing 1.864g of KCl (AR). Potassium chloride of varying concentrations of (0.05N, 0.04N, 0.03N, 0.02N, 0.01N, 0.005N, 0.0025N and 0.001N) were prepared from 0.1N KCl. The clean conductivity cell was first rinsed with 0.05N potassium chloride and required volume of KCl (0.05N) was taken in a dry beaker. The conductivity cell was kept inside the beaker. The specific conductivity of the above solution was measured. The measurements were repeated for other solution after rinsing each time with the test solution. The equivalent conductance values were determined by using the formula,

$$\lambda_c = k \times \frac{1000}{C}$$

where,

k is specific conductance, C is concentration of the solution.

RESULT :

As dilution increases equivalent conductance _____.

$\lambda = 530\text{nm}$

Strength of KMnO_4 from which dilution is made = 0.001M

S. No.	Concentration of $\text{KMnO}_4 \times 10^{-4}\text{M}$	Volume of 0.001M KMnO_4 (ml)	Absorbance
1			
2			
3			
4			
5			
6			
7			
8			
9			

Fit $\lambda = 420\text{nm}$

Strength of K_2CrO_4 from which dilution is made = 0.02M

S. No.	Concentration of $\text{K}_2\text{CrO}_4 \times 10^{-3}\text{M}$	Volume of $0.02\text{M K}_2\text{CrO}_4$ (ml)	Absorbance
1			
2			
3			
4			
5			
6			
7			
8			
9			

COLORIMETRY

AIM :

To test the validity of Beer – Lambert’s law using photo electric colorimeter and to determine the unknown concentration of the given solution.

PRINCIPLE :

According to Beer – Lambert’s Law $A = \epsilon C \ell$

where,

A = absorbance or optical density

ϵ = molar absorption coefficient

C = Concentration of absorbing solution is mol/dm³

ℓ = Length of the cell

The Beer-Lambert’s law may be verified the calibration curve obtained. The concentration of the unknown solution can be determined by measuring its absorbance.

PROCEDURE :

A stock solution of 0.001M KMnO₄ and 0.02M K₂CrO₄ prepared and is diluted quantitatively to the required range. By using appropriate filter (KMnO₄ = 52, K₂CrO₄ = 43), the absorbance of the above solution were measured. Absorbance was plotted against concentration. The absorbance of the given unknown solution were measured corresponding to this optical densities, the concentration of unknown solution can be determined from the calibration curve.

RESULT :

Linear relationship between absorbance and concentration indicates the validity of Beer – Lambert’s law.

(i) The concentrations of unknown potassium permanganate solution are

(a) (b)

(ii) The concentrations of unknown potassium chromate solutions are

(a) (b)

ROUGH TITRATION :

Volume of NaOH (ml)	emf value (V)	emf value (mV)	ΔE (mV)

FAIR TITRATION :

Volume of NaOH (ml)	emf value (V)	emf value (mV)	ΔE in (mV)	Δv in (ml)	$\Delta E / \Delta V$ in (mV/ml)	Mean volume of NaOH (ml)

Calculation :

Volume of hydrochloric acid (V_1) =

Strength of hydrochloric acid (N_1) =

Volume of Sodium hydroxide (V_2) =

Strength of Sodium hydroxide (N_2) = $\frac{V_1 N_1}{V_2}$

Strength of Sodium hydroxide = N

POTENTIOMETRY

ACID – BASE TITRATION

AIM :

To determine the concentration of sodium hydroxide using standard hydrochloric acid by potentiometric titration.

PROCEDURE :

Accurately 0.05N HCl acid was prepared 10ml of this was pipetted out into a beaker equal amount of distilled water was added. A pinch of quinhydrone was added. The platinum electrode was dipped into a solution and was connected to positive terminal and the reference electrode also dipped into the solution and was connected negative terminal of the potentiometer. Then the given sodium hydroxide was taken in burette and added to the solution in the beaker. After each addition the emf values and range of equivalence point was noted. An accurate experiment was carried out by noting the emf value for each addition of 1cc portion, 0.5cc, 0.2cc and 0.1cc portion of the titrant were added.

The following graph were drawn

- (1) emf Vs volume of sodium hydroxide
- (2) $\Delta E / \Delta V$ Vs mean volume of sodium hydroxide

RESULT :

The strength of given sodium hydroxide solution = N

ROUGH TITRATION :

Volume of KMnO ₄ (ml)	emf value in (V)	emf value in (mV)	ΔE in (mV)

FAIR TITRATION :

Volume of KMnO ₄ (ml)	emf value (V)	emf value (mV)	ΔE (mV)	Δv in (ml)	ΔE / ΔV in (mV / ml)	Mean value of KMnO ₄ (ml)

Calculation :

Volume of Ferrous Ammonium Sulphate (V₁) =

Strength of Ferrous Ammonium Sulphate(N₁) =

Volume of Potassium permanganate (V₂) =

Strength of Potassium permanganate (N₂) = $\frac{V_1 N_1}{V_2}$

Strength of Potassium permanganate = N

POTENTIOMETRY

REDOX TITRATION

AIM :

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

PROCEDURE :

Accurately 0.05N KMnO_4 solution was prepared and 10ml of FAS was pipetted out into a beaker, equal amount of dilute sulphuric acid was added. A platinum electrode was dipped into the solution and was connected to positive terminal and the reference electrode also dipped into the solution and was connected to the negative terminal of the potentiometer then the given KMnO_4 was taken in burette and added to the solution in the beaker. For each addition of KMnO_4 the corresponding emf value was noted. From the emf value, the range of equivalent point was noted. At the equivalence point 0.5cc, 0.2cc and 0.1cc portion of the titrant were added.

The following graph were drawn

- (1) emf Vs volume of KMnO_4
- (2) $\frac{\Delta E}{\Delta V}$ Vs mean volume of KMnO_4

RESULT :

The strength of the given potassium permanganate solution is = N

