THIRUTHANGAL NADAR COLLEGE

(Belongs to the Chennaivazh Thiruthangal Hindu Nadar Uravinmurai Dharma Fund)

Selavayal, Chennai-51.

A Self-Financing Co-educational College of Arts & Science

Affiliated to the University of Madras

Accredited with ‘B’ Grade by NAAC

An ISO 9001: 2015 Certified Institution

NAME OF THE DEPARTMENT: CHEMISTRY

SUBJECT : PHYSICAL CHEMISTRY PRACTICAL

TOPIC : Experiment Numbers 1 to 6

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Syllabus

Physical Chemistry Experiments

1. Critical solution temperature
2. Effect of temperature on critical solution temperature
3. Rast method
4. Transition temperature
5. Heat of neutralization
6. Phase diagram(Simple Eutectic)
7. Kinetics of iodination of acetone
8. Kinetics of eater hydrolysis
9. Kinetics of Persulphate-iodide reaction
10. Viscosity
11. Partition coefficient and equilibrium constant of KI+I->KI
12. Determination of cell constant, specific conductance and equivalent conductance of strong electrolyte.
13. Conductometric acid-Base titration
14. Conductometric Precipitation titration
15. Potentiometric acid-Base titration
16. Potentiometric redox titration

1. Critical solution temperature

Theory

When two liquids mixed,

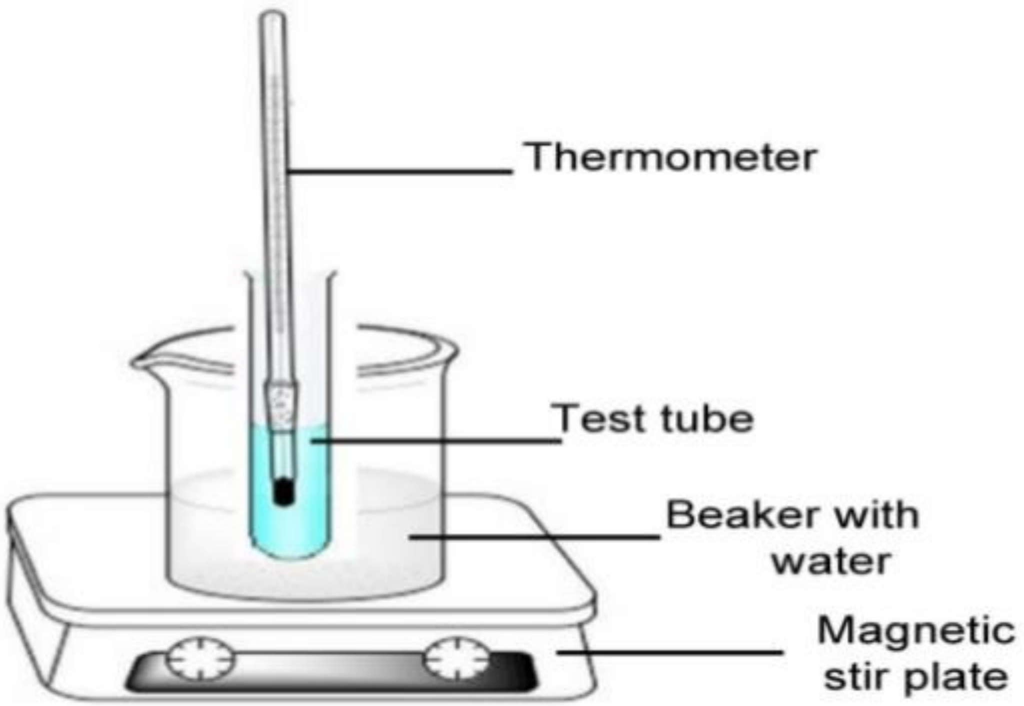
(i) completely miscible

(ii) completely immiscible

(iii) partially miscible

Phenol-water forms partially miscible mixture

Critical solution temperature…



Diag. experimental arrangement for cst determination of phenol water system

1. Critical solution temperature…

- Procedure:- 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.

- 4)At a certain temperature, the mixture becomes **clear**. Note this temperature (t_1˚C).

  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** (t_2˚C).

  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.*

Amount of oxalic acid = 1.32 g

$$\text{Normality} = \frac{weight}{eq.wt} \times \frac{1000}{V}$$

$$= \frac{1.60}{63} \times \frac{1000}{250} = 0.101 \text{ N}$$

Standardization of oxalic acid

Sl. No	Volume of NaOH Solution (mL)	Burette readings		Volume of Oxalic acid Solution (mL)	Indicator
		Initial (mL)	Final (mL)		
1.					
2.					

Concordant Value = ______ mL

Calculation:

Volume of oxalic acid solution V_1 = _____ mL

Strength of oxalic acid solution N_1 = _____ N

Volume of NaOH solution V_2 = _____ mL

Strength of NaOH solution N_2 = ? N

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

Strength of NaOH solution = ______ N

1. Conductometric Acid-Base Titration

Aim

To estimate the amount of acid present in a given solution conductometrically. You are provided a decinormal solution of NaOH.

Apparatus and Chemicals Required:

- Conductivity meter
- Conductivity cell
- NaOH solution
- HCl solution
- Beaker
- Burette
- Pipette
- Standard measuring flask

Principle:

The conductivity of the solution is related to the mobility of ions which in turn related with the size of the ions. When the titration of strong acid and strong base are carried out, there is a decrease in conductivity as highly mobilized hydrogen ions are replaced by sodium ions.

$$NaOH + HCl \rightarrow NaCl + H_2O$$

After the acid is consumed, there is a steep increase in conductivity which gives the end point and this increase in conductivity is due to the fast moving hydroxyl ions from the base. From this end point, the amount of acid present is calculated.

Procedure:

Preparation of Standard Oxalic Acid Solution:

Equivalent weight of $H_2C_2O_4.H_2O$ is 63 and hence accurately 1.60 g of oxalic acid crystals is carefully weighed and dissolved in distilled water in a 250 mL standard measuring flask. It is made up to the mark using distilled water.

Estimation of HCl

S. No.	Volume of NaOH (mL)	Conductance (mho)

Volume of NaOH Solution V_1 = ______ mL

Strength of NaOH Solution N_1 = ______ N

Volume of HCl Solution V_2 = _____ mL

Strength of HCl Solution N_2 = ? N

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

Strength of HCl Solution = ________ N

Amount Calculation:

Amount of HCl present in the whole of the given solution $= \frac{V \times E \times N}{1000}$

Standardization of NaOH

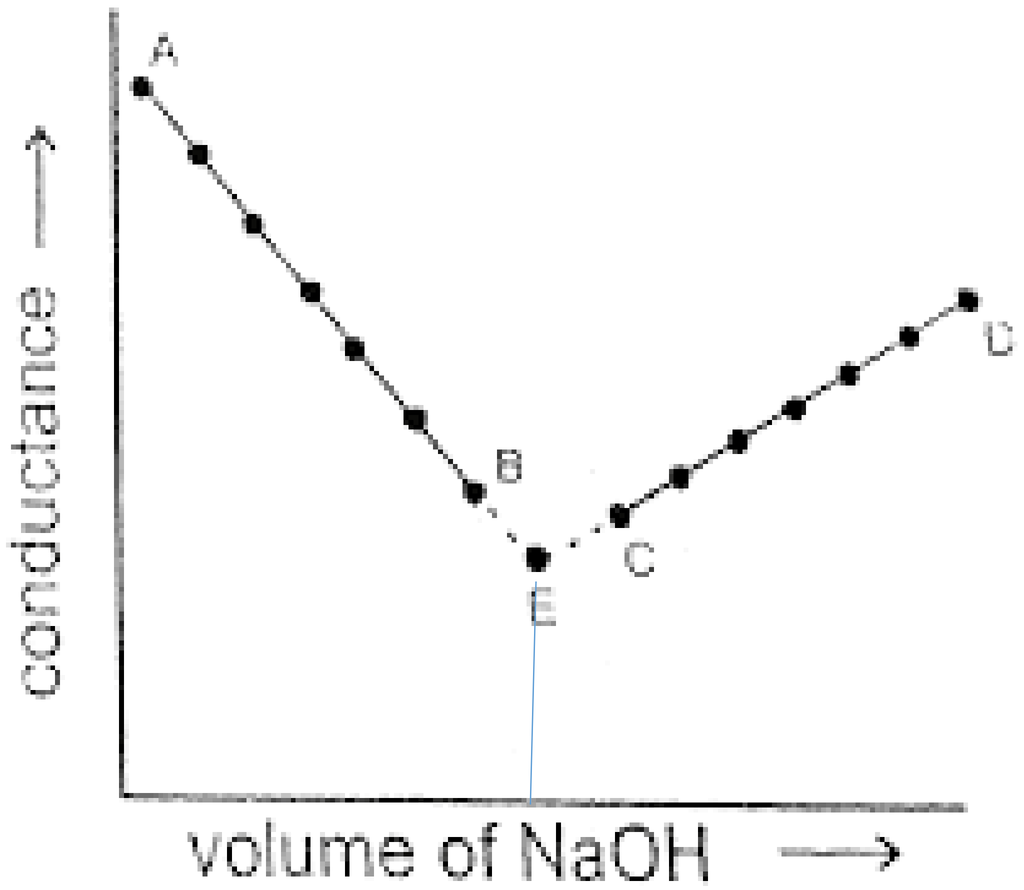
The burette is washed with distilled water and rinsed with NaOH solution. Then it is filled with NaOH solution up to the zero mark. Exactly 20 mL of standard oxalic acid solution is pipetted out using a rinsed pipette in to a clean conical flask. To this solution, 2-3 drops of phenolphthalein indicator is added which turns the solution into pink colour. This solution is titrated against NaOH solution taken in the burette. The end point is disappearance of pink colour. The titration is repeated to get concordant values. From the titre value, the strength of NaOH solution is determined.

Estimation of HCl:

The given mixture of acids is made up to 100 ml using distilled water. 10 ml of this made up solution is pipette out into clean beaker and 100 ml of distilled water is added. The conductivity cell is dipped into the test solution and the base NaOH is added in an interval of 0.5 ml with uniform stirring. The conductance is measured after each addition of NaOH at various stages of neutralization. After complete neutralization, the amount of acid present in the given mixture is determined based on the volume of base consumed. Volume of base consumed for strong acid and weak acid are determined by plotting a graph between corrected conductance and volume of base added, where first end point corresponds to strong acid and second end point corresponds to weak acid.

Result:

The amount of HCl present in the whole of the given solution is ________ g


A
B
E
C
D
conductance
volume of NaOH

2. Critical Solution Temperature

Aim

To determine critical solution temperature and critical solution composition of phenol-water system and to find the composition of the given unknown mixture

Apparatus and Chemicals Required

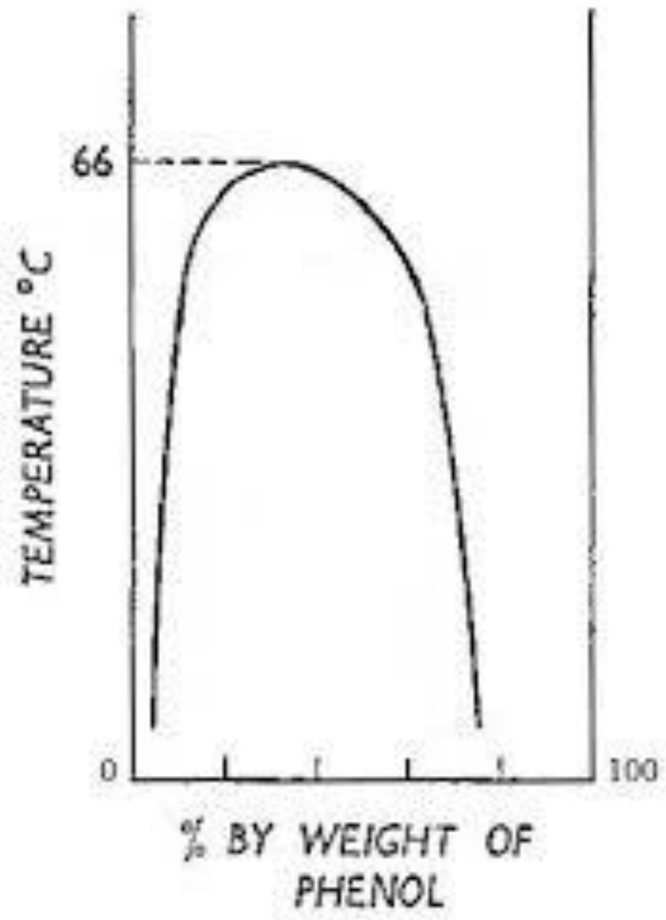
- Graduated pipette
- TT Apparatus
- Thermometer
- Water bath
- Phenol
- Distilled water

Principle

Phenol and water belong to a system of partially miscible liquid which is indicated by a gelatinous white turbidity. The miscibility of this mixture increases with increase of temperature. The temperature at which the turbidity disappears is called the miscibility temperature.

In this experiment miscibility temperatures of different mixtures of phenol and water are determined. When the miscibility temperature is plotted against percentage composition of mixture, a graph with inverted 'U' shape curve is obtained. Temperature corresponding to the highest point of the curve gives the critical solution temperature and critical solution composition of the system.

The temperature above which the two partially miscible liquids become completely miscible at all proportions is called critical solution temperature.


66
TEMPERATURE °C
0
100
% BY WEIGHT OF PHENOL

S. No.	Volume of Phenol (mL)	Volume of water (mL)	Weight of phenol (g)	Weight of water (g)	% of Phenol	Miscibility temperature (°C)
1	0.7	4.3	0.749	4.3	14.8	60.0
2	1.0	4.0	1.07	4.0	21.1	61.5
3	1.5	3.5	1.605	3.5	31.4	64.0
4	2.0	3.0	2.14	3.0	41.6	63.8
5	**2.5**	**2.5**	**2.675**	**2.5**	**51.7**	**63.0**
6	3.0	2.0	3.21	2.0	61.6	59.0
7	3.5	1.5	3.531	1.5	70.1	54.0
8	Unknown	-	-	-	-	62.3

$$\text{weight of phenol} = \text{volume} \times \text{density}$$

$$\text{weight of water} = \text{volume} \times \text{density}$$

$$\text{percentage of phenol} = \frac{\text{weight of phenol}}{\text{weight of phenol} + \text{weight of water}} \times 100$$

Result

1. The critical solution temperature of phenol-water system is _________
2. The critical solution composition of phenol water system is _________

3. Effect of impurities on critical solution temperature

Aim

To study the effect of an impurity (sodium chloride) on the CST of the phenol-water system and to determine the concentration of NaCl in an unknown solution by critical solution temperature method.

Apparatus and Chemicals Required

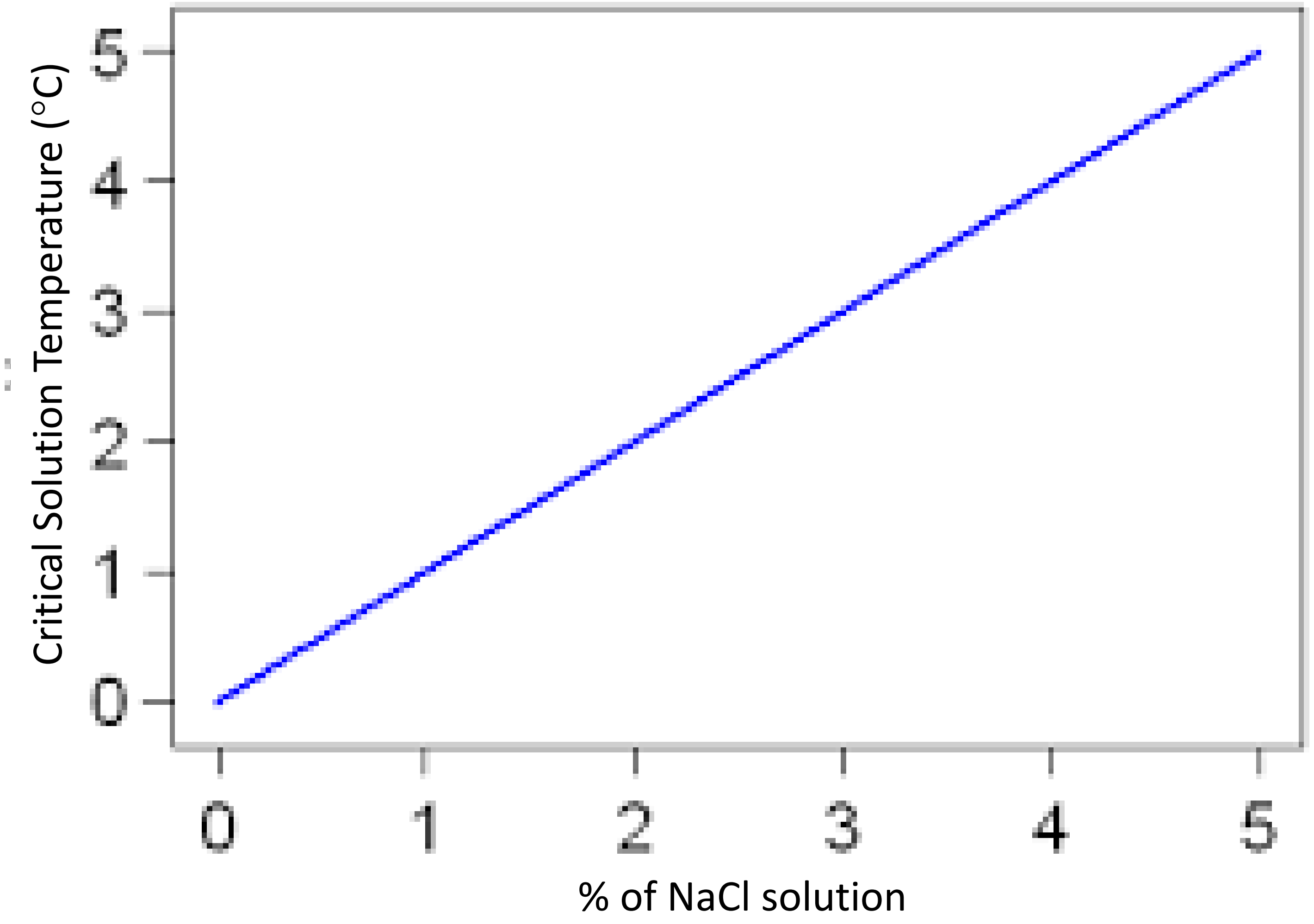
- Graduated pipette
- TT Apparatus
- Thermometer
- Water bath
- Phenol
- Distilled water
- 1% NaCl solution

Principle

Sodium chloride is soluble only in water and not in phenol. Therefore, it will raise the CST of phenol-water system. In this experiment, the miscibility temperatures for various mixtures of phenol and aqueous solutions of sodium chloride are determined. A calibration curve (usually a straight line) is plotted between the concentration of NaCl and the miscibility temperature. The miscibility temperature for a mixture of phenol and the unknown NaCl solution is also determined. From the calibration curve, the concentration of NaCl solution is then obtained.

Table

S. No.	Volume of Phenol (mL)	Volume of 1% NaCl solution (mL)	Volume of distilled water (mL)	% of NaCl in aqueous solution	Miscibility temperature (°C)
1	2.5	0	2.5	0	63.0
2	2.5	0.5	2.0	0.2	65.2
3	2.5	1.0	1.5	0.4	67.0
4	2.5	1.5	1.0	0.6	68.7
5	2.5	2.0	0.5	0.8	70.6
6	2.5	2.5	0	1.0	71.8
7	2.5	2.5 (unknown)	-		68.2


Critical Solution Temperature (°C)
0
1
2
3
4
5
0
1
2
3
4
5
% of NaCl solution

Result

The Concentration of NaCl solution is _________

4. Determination of molecular weight by Rast method

Aim

To determine k_f of naphthalene and hence to find the molecular weight of the given unknown substance

Apparatus and Chemicals Required

- TT apparatus
- Sensitivity thermometer
- Stirrer
- Water bath
- Naphthalene
- Reference substance
- Unknown substance

Principle

Naphthalene mixed homogeneously with an inert solid substance forms a solid solution which freezes at a lower temperature than the normal freezing point of pure naphthalene. In case of dilute solution, the depression of freezing point is given by,

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

$\Delta T =$ depression in the freezing point

$K_f =$ molal depression constant of the solvent (naphthalene)

$W_2 =$ weight of the solute

$W_1 =$ weight of the solvent

$M_2 =$ molecular weight of the solute

From the freezing point measurement, the K_f of naphthalene can be found using a a reference substance. From the K_f value, the molecular weight of the unknown substance can be determined.

Calculation

Determination of melting point of naphthalene

Melting point of naphthalene =

Determination of K_f

The given reference substance is ___________

Molecular weight of the reference substance M_2 =

Weight of the reference substance W_2 =

Weight of naphthalene W_1 =

Melting point of the reference substance in naphthalene =

ΔT = _______ - _________ = ________ °C

The Kf of naphthalene is,

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

=

Calculation…

–

Determination of molecular weight,

Weight of naphthalene W_1 =

Weight of the unknown substance W_2 =

Melting point of the unknown substance in naphthalene =

ΔT = ________ - __________ = __________°C

The molecular weight of the given unknown substance is,

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

=

Result:

The Kf of naphthalene =

The molecular weight of the given substance =

5. Determination of transition temperature

Aim

To determine the transition temperature of the given salt hydrate by thermometric method

Apparatus and Chemicals Required

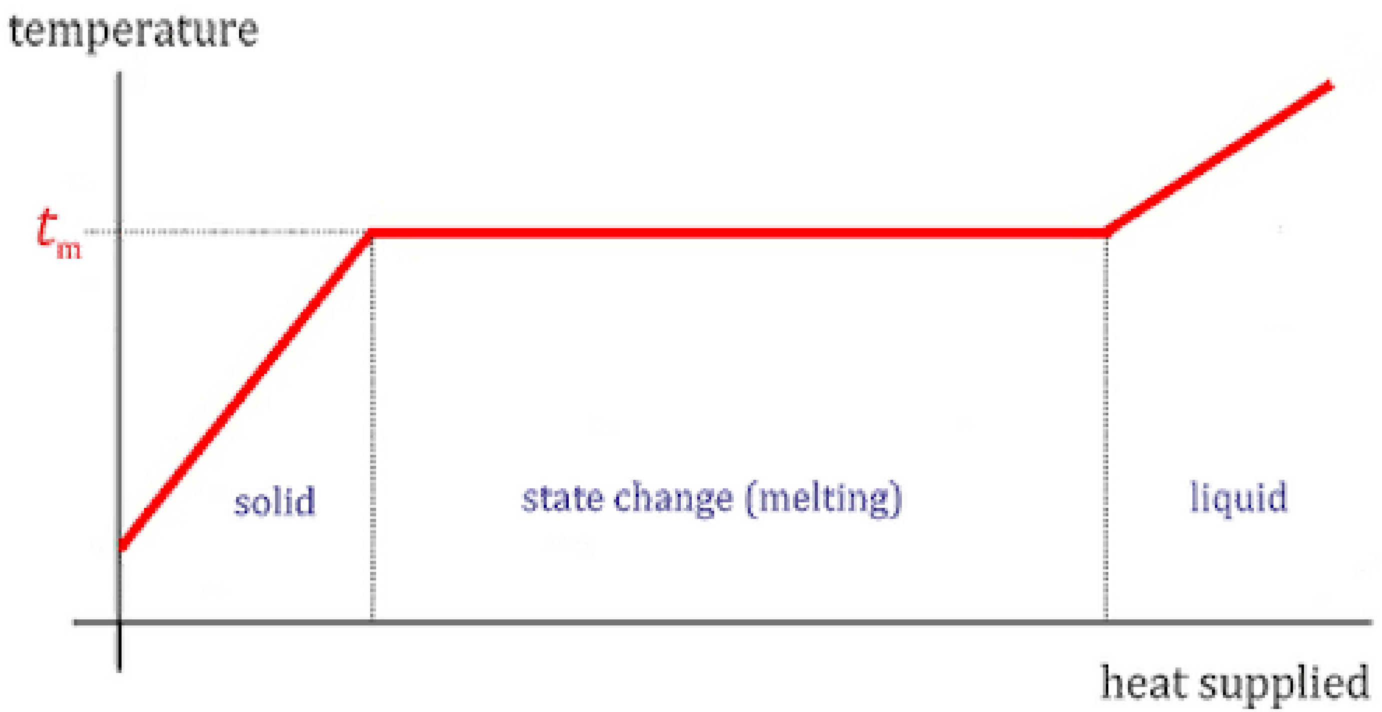
- TT apparatus
- Sensitivity thermometer
- Stirrer
- Water bath
- Sodium thiosulphate pentahydrate

Principle

Many of the crystalline hydrated salt on heating loss their water of crystallization and change to an anhydrous form or to less hydrated crystalline form. During this change, the temperature of the system remains constant. This constant temperature is caused by the transition of salt hydrate. For example, sodium thiosulphate pentahydrate system undergoes the following transition,

$$Na_2S_2O_3.5H_2O \xrightarrow{heat} Na_2S_2O_3 + 5H_2O$$

The salt hydrate is heated over a water bath and temperature is measured with time interval. A graph is plotted between temperature with time. The transition temperature can be determined from the graph.


temperature
t_m
solid
state change (melting)
liquid
heat supplied

Heating		Cooling	
Time (s)	Temperature (°c)	Time (s)	Temperature (°C)

Result:

The transition temperature of the given salt hydrate is =

6. Phase diagram (Simple eutectic)

Aim

To study the simple eutectic system formed by naphthalene and *p*-nitrotoluene and hence to determine the eutectic composition, eutectic temperature and composition of unknown

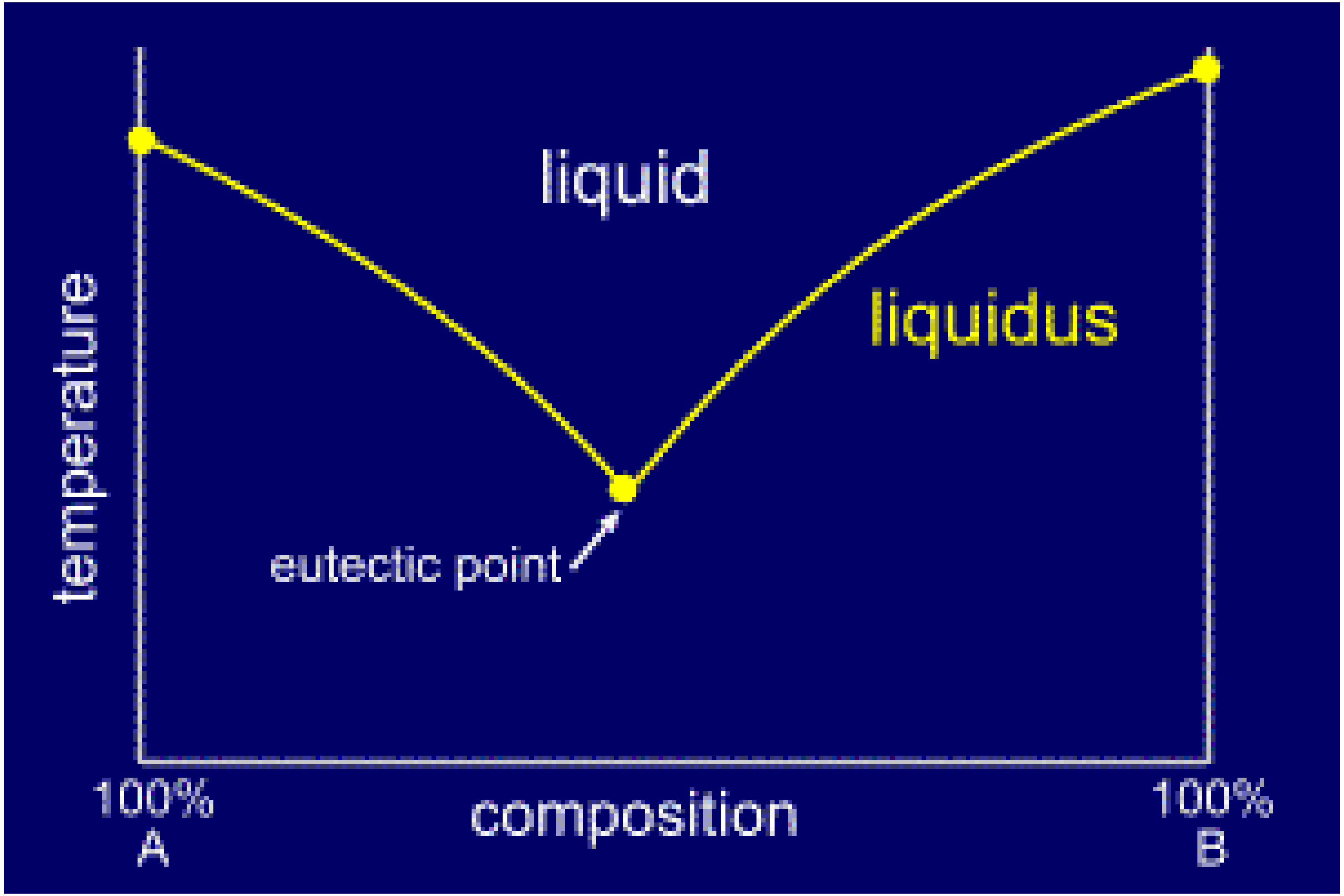
Apparatus and Chemicals Required

- TT apparatus
- Sensitivity thermometer
- Stirrer
- Water bath
- Naphthalene
- *P*-Nitrotoluene

Principle

When p-nitrotoluene is added to naphthalene, it depresses the freezing point of naphthalene. For successive additions of p-nitrotoluene, the freezing point of resulting solution goes on decreasing. Similarly when naphthalene is added to p-nitrotoluene, it depresses the freesing point of p-nitrotoluene.

When freezing point is plotted against the percentage of composition of p-nitrotoluence, simple eutectic system graph is obtained. In graph, point E is called the eutectic point. Eutectic means 'easy melting'. The composition and temperature corresponding to point E are known as eutectic composition and eutectic temperature.


liquid
liquidus
temperature
eutectic point
100%
A
composition
100%
B

Weight of naphthalene = 2.0 g

S. No.	Weight of p-nitrotoluene added	Total weight of p-nitrotoluene	Weight percentage of p-nitrotoluene	Freezing point (°C)
1	0	0	0	
2	0.22	0.22	9.99	
3	0.28	0.50	20.0	
4	0.36	0.86	30.1	
5	0.47	1.33	39.9	
6	0.67	2.00	50.0	
7	1.00	3.00	60.0	

Weight of p-nitrotoluene = 2.0 g

S. No.	Weight of naphthalene added	Total weight of Naphthalene	Weight percentage of p-nitrotoluene	Freezing point (°C)
1	0	0	100.0	
2	0.22	0.22	90.1	
3	0.28	0.50	80.0	
4	0.36	0.86	69.9	

Result:

1. Eutectic composition is _________ % of p-nitrotoluene
2. Eutectic temperature is ___________
3. Amount of p-nitrotoluene in the given mixture is ________%