

KING SAUD UNIVERSITY

College of Science

Chemistry department



CHEM336

PHYSICAL CHEMISTRY OF SOLUTIONS (Laboratory)

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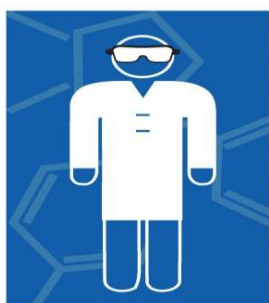
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Course grades distribution:

The total score of the practical part is 30 points, distributed as follows:

1. Attendance and reports (10 points)
2. First exam(5 points)
3. Final exam (15 points)

Laboratory Safety and Guidelines:



Lab Safety Rules

Science labs offer great opportunities for learning, teaching, and research. They also pose hazards that require proper safety precautions.



Sources: Carolina Biological Supply Company. "Lab Safety Dos and Don'ts for Students." <http://www.carolina.com/teacher-resources/interactive/lab-safety-instructions/tr11076.tr>



Dress appropriately

Tie back long hair, and wear suitable gloves, goggles, and other protective equipment.

Proper supervision

Don't perform lab experiments without instructor supervision (unless given permission to do so).



Know location of emergency numbers & safety equipment

Know the location of safety equipment and emergency phone numbers (such as poison control) so you can access them quickly if necessary.



No food

Don't eat or drink in the lab—and never taste chemicals.



ID hazards

Identify hazardous materials before beginning labs.



Be attentive

Be attentive while in the lab. Don't leave lit Bunsen burners unattended or leave an experiment in progress.

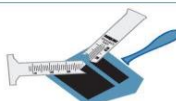
Be careful when handling hot glassware

Turn off all heating appliances when not in use. Keep flammable objects away from your workspace.



Keep a clean workspace

Don't obstruct work areas, floors, or exits. Keep coats, bags, and other personal items stored in designated areas away from the lab. Don't block sink drains with debris.



Handle glassware carefully

Properly dispose of anything that breaks. Report cuts, spills, and broken glass to your instructor immediately.



Clean up

After completing the lab, carefully clean your workspace and the equipment, and wash your hands.

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Calculations and Presentation of Data

1) Graphs and Graphical Methods

By a graph we mean a representation of numerical values or functions by the positions of points and lines on a two-dimensional surface. A graph is inherently more limited in precision than a table of numerical values or an analytic equation, but it can contribute a "feel" for the behavior of data and functions that numerical tables and equations cannot. A graph reveals much more clearly such features as linearity or nonlinearity, maxima and minima, points of inflection, etc.

In preparing final plots, whether computer generated or prepared by hand, there are several important features to consider:

1. Choose the Graph Paper and Indicate the Axes. Graph paper is available with linear, semilog, and log-log scales. Most good graph paper is ruled in soft colors that do not distract the viewer from the plotted points and lines. Indeed, the presence of a lined grid makes it very easy to "read" values from the plot (say

interpolate between points to find y at some desired value of x). In choosing the horizontal and vertical scales to be used, attention should be paid to making a commensurate choice where a major scale division corresponds to an attractive integral number of the smallest divisions.

2. Use Clear Scale Labels. Choose suitable scale divisions with longer tick marks used for the major values along a scale that will be labeled with the appropriate numerical values. Under the scale numbers for the independent variable, enter the scale label, stating the quantity that is varying and its units: e.g., T(K) for absolute temperature in kelvin. To avoid scale numbers that are too large or too small for convenient use, multiply the quantity by a power of 10.
3. Distinguish Smooth Curves. If your plot contains several empirical smooth curves or several theoretical curves, it is wise to distinguish them by using dashed and dash-dot curves as well as solid lines.
4. Add a Legend. Somewhere on the figure (if possible, at the bottom) enter a legend with an identifying figure number. The legend should state the contents of the figure, identify the symbols and line types used, and provide any needed information that will not be provided in the text of the document in which the figure will appear.
5. Plot the Points and Draw the Lines and Curves. Using a sharp, hard pencil, plot the experimental points as small dots, as accurately as possible. Draw small circles (or squares, triangles, etc.) of uniform size (2 or 3 mm) around the points in ink to give them greater visibility.

Experiment No (1)

Determination of partial molar volume

Objective:

To calculate partial molar volumes of water-ethanol mixtures as a function of concentration from densities measured with a pycnometer.

Theory:

the **extensive properties** (such as volume, free energy, enthalpy, etc.) of a solution are not additive properties of its components. For example, when one mixes 1 mole of H₂O (which has a molar volume of 18 cm³) with a large quantity of ethanol, the volume increase observed is not 18 cm³. The surrounding of a molecule is very important in determining how much volume it will occupy, how much energy it will have, etc. So, H₂O molecules surrounded by other H₂O molecules behaves quite differently from H₂O molecules surrounded by ethanol molecules.

Partial molar volume \bar{V}_i : The change in molar volume V when one mole of A_i is added to an “infinitely large” sample at constant T and P .

$$\bar{V}_A = \left(\frac{\partial v_T}{\partial n_A} \right)_{T,P,n_B} \quad (1)$$

Equation 1 gives the partial molar volume of the component A for a binary system with components A and B. Where V is the total volume, n_A is the number of moles of A.

Experimental Determination of Partial Molar Volume using tangent-intercept method

The total volume of the binary solution composed of A (“solvent”) and B (“solute”) becomes

$$V_T = n_A \bar{V}_A + n_B \bar{V}_B \quad (2)$$

It is convenient to express this on a per-mole basis, dividing through by the total number of moles ($n_A + n_B$) to obtain the equivalent equation in terms of mole fraction X_B (with $X_A = 1 - X_B$)

$$\bar{V} = X_A \bar{V}_A + X_B \bar{V}_B = (1 - X_B) \bar{V}_A + X_B \bar{V}_B = \bar{V}_A + X_B (\bar{V}_B - \bar{V}_A) \quad (3)$$

As shown by the final equation on the right, the equation for $\bar{V} = \bar{V}(X_B)$ is that of a straight with slope

$$\frac{d\bar{V}}{dX_B} = \bar{V}_B - \bar{V}_A \quad (4)$$

And intercept \bar{V}_A . The graph below shows the plotted behavior of $V_{(X_B)}$ (solid line) from \bar{V}_A^0 (at $X_B = 0$) to \bar{V}_B^0 (at $X_B = 1$), with its corresponding tangent line (dashed) at the concentration of interest (circle, dotted line). As shown in the graph, extrapolation of the tangent line to the axes yields \bar{V}_A (on the left) and \bar{V}_B (on the right). The extrapolated-tangent construction therefore makes it easy to read off the partial molar volumes \bar{V}_A , \bar{V}_B at any desired concentration X_B .

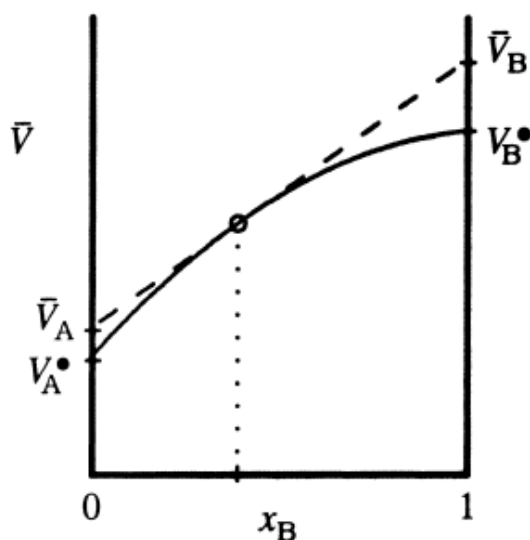


Figure 1

Chemicals and Apparatus

Ethanol, distilled water, 25 ml pycnometer, balance, graduated cylinder, flask...etc.

Procedure

- Prepare a series of solutions by weight containing ethanol and water 20, 40, 60, 80-wt% of ethanol.
- Determine the density of each solution at room temperature using the following procedure:
 - Determine the weight of previously oven-dried and cooled pycnometer (m_1).

- Fill up the empty pycnometer with distilled water, making sure that the water level in the pycnometer reaches the top of the capillary and is free of air bubbles.
- Determine the weight of the pycnometer, which is filled with water $m_{(\text{Pyc}+\text{water})}$. When doing this, make sure that the outside of the pycnometer is completely dry.
- Given the density of water at ambient temperature, $\rho = 0.997 \text{ g/ml}$ calculate the volume of pycnometer using equation 5.

$$V_{(\text{Pyc})} = \frac{m_2 - m_1}{\rho} \quad (5)$$

- Subsequently weigh the pycnometer, which is filled with different mixtures of ethanol and water (m_2) then calculate the densities of these solutions.
- Calculate the specific volume d^{-1} (= reciprocal density ml/g) for each solution and plot the specific volume versus mole fraction of ethanol for each solution.
- Draw a smooth curve through all points.
- Draw tangent lines to this curve at different concentrations and determine the y-intercepts of these lines on $X=0$ and $X=1$ the former is the specific volume of water and the latter is the specific volume of ethanol.
- Calculate the partial molar volume of each component using equation 6.

$$\bar{V} = M_{\text{wt}} d^{-1} \quad (6)$$

M_{wt} of ethanol = 46.07 g/mol and M_{wt} of water = 18.015 g/mol

- Draw two “partial molar volume versus mole fraction” curves for water and ethanol and determine the partial molar volumes for the components at $X_{(\text{EtOH})} = 0.2, 0.4, 0.6,$ and 0.8 .
- Calculate the total volume of each solution using equation (2).

Report

Students' Names:

Object:

m_1 (empty Pyc) =	m (Pyc + water) =	$V_{(pyc)}$ =
---------------------	---------------------	---------------

W%	$m = m_2 - m_1$	d^{-1}	$\bar{V}_{(EtOH)}$	$\bar{V}_{(water)}$	V_T
0					
20					
40					
60					
80					
100					

1. Plot the specific volume versus mole fraction of ethanol for each solution.
2. Plot two “partial molar volume versus mole fraction” curves for water and ethanol

Experiment No (2-3)

Raoult's Law for Ideal Solutions

Object: to examine how two different mixtures (hexane-heptane and hexane-isopropanol) obey Raoult's law.

Introduction: The concept of an ideal solution is important in the development of an understanding of the properties of real solutions. In a liquid solution, molecules are in intimate contact with one another so that the question of ideality is determined by the nature of the intermolecular forces. Suppose a solution is formed by mixing two liquids, A and B. Then, the solution is ideal if the intermolecular forces between A and B molecules are no different from those between A and A, or B and B molecules. An indication of whether or not the condition for ideality is met is obtained from the vapor pressure of the solution. At a given temperature, the vapor pressure of a pure liquid is a measure of the ability of molecules to escape from the liquid to the gas phase. By studying the vapor pressure of a solution as a function of its composition at constant temperature one may assess the solution's ideality or its degree of departure from ideality. For an ideal solution, the tendency of molecule A to escape is proportional to its mole fraction, that is, to its concentration expressed in terms of the fraction of molecules which are of type A. The proportionality constant must be the vapor pressure of pure component A because this vapor pressure is reached when the mole fraction is unity. This result is **Raoult's law**, which is expressed mathematically as

$$P_A = P^\circ_A X_A \quad (1)$$

Similarly, for the other component B,

$$P_B = P^\circ_B X_B \quad (2)$$

The total pressure above the solution includes both components; the solvent and the solute unless the solute is nonvolatile.

$$P_T = P^\circ_A X_A + P^\circ_B X_B \quad (3)$$

When a solution is boiling, its vapor pressure (P_T) is equal to the current atmospheric pressure. To assess the solution's ideality, we need to measure both P (solvent) and P (solute) at the boiling temperatures. Although it is relatively straightforward to

determine the composition of the solution phase, measuring the composition of the vapor phase is a bit more difficult. However, it's possible to calculate vapor pressure of a pure liquid (The proportionality constant in Raoult's law) using the Clausius-Clapeyron equation.

Clausius-Clapeyron equation (4), although only approximate, provides a useful tool for estimating the vapor pressure changes of a pure liquid as a function of temperature.

$$\ln \left(\frac{P_{vap}}{P^\circ} \right) = \frac{-\Delta H_{vap}(T,P)}{R} \left(\frac{1}{T(K)} - \frac{1}{T(K)_{bp}} \right) \quad (4)$$

$$P_{vap} = e^{\frac{-\Delta H_{vap}}{R} \left(\frac{1}{T(K)} - \frac{1}{T(K)_{bp}} \right)}$$

Where P° is the atmospheric pressure which is equal to 1, P is the vapor pressure at some absolute temperature T , R is the universal gas constant and T_{bp} is pure liquid's normal boiling point. $\Delta H_{vap}(T, P)$ is the heat of vaporization, it is function in temperature and pressure, but it is nearly constant over the usual effective range of T and P variations, we assume that it can be replaced by the fixed molar enthalpy of vaporization ΔH°_{vap} for standard state conditions (e.g., 25 C, 1 atm).

Condenser is open here; do *not* cork!

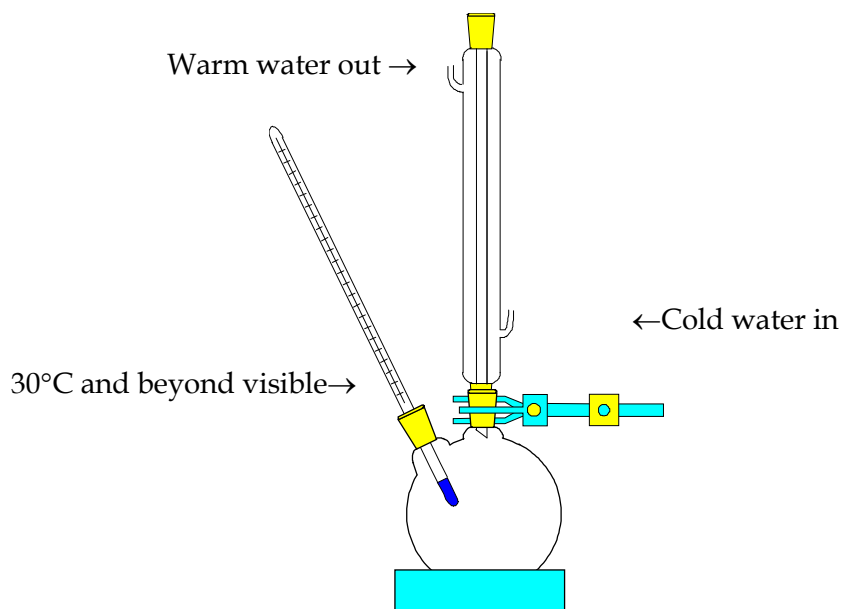


Figure 2

Chemicals and Apparatus

3-neck 19/22 round bottom flask, 19/22 condenser with cold water tubing, C-clamp, 0.1°C thermometer, 10 ml pipette, heating mantle, boiling beads, and stopcock grease. Pentane, isopropanol and heptane or hexane.

Procedure

1. Read the description and instructions carefully in the technique part and construct a boiling point apparatus from the flask, condenser, thermometer, tubing, and heating mantle as shown in Fig.1.
2. Add 50 ml of **hexane** to the round-bottom flask. How many moles is that? Heat the flask and reflux the contents for about 7-10 minutes until the equilibrium is established and record the equilibrium temperature as the boiling point of the liquid.
3. Pipette **ten** (5-ml) aliquots of **heptane** into the flask, record the moles and note the boiling point of the mixture after each addition.
4. Repeat the experiment with fresh pentane solvent but use isopropanol as solute.
5. Calculate number of moles (n) and moles fraction (X) of each solution (While you add solutions by volume, and you know the densities of pure liquid so you can determine the additional masses. From those masses you can calculate mole and mole fraction for each component)
6. Using the Clausius-Clapeyron equation (2) to calculate the vapor pressure P° above each pure liquid for each measured T.
7. Determine to what extent these mixtures obey Raoult's law. (when the mixture is boiling, its vapor pressure is equal (the current) atmospheric pressure (supposed to be 1atm) and to the extent to which Raoult's Law is obeyed, $P^\circ_A X_A + P^\circ_B X_B$ will be the mixture's vapor pressure, the current atmospheric pressure). In other words, to what extent $P^\circ_A X_A + P^\circ_B X_B$ is equal to unit

hint 1: Use the following physical data

Reagent	T [°] _{bp} °C	Density (g/ml)	Δ H _{vap} at 25°C, kJ/mol
Pentane	36.06	0.626	26.4
Heptane	98.5	0.684	36.57
Isopropanol	82.3	0.785	45.39
Hexane	68	0.659	31.73

Technique: We will use a procedure called **reflux**. As the solution boils, cool water in the condenser will condense the vapor and the liquid will re-enter the flask. You should smell little or no vapor during the experiment.

Using a clamp, attach a round-bottom flask with a condenser to a burette stand. The flask should rest on a heating mantle on a **heat-resistant surface**. Drop a few boiling beads into the flask. Smear a small amount of stopcock grease on the tip of the thermometer to make it slide more easily into its cork. Insert the thermometer into the flask's side arm so that it does not touch the flask's sides or bottom. Be sure you can read the thermometer from 30°C. The bulb should be inside the vapor as the solution boils; you'll see *condensation forming on it*; that ensures it is measuring the boiling point accurately. The thermometer should not be **IN** the liquid!

Attach tubing snugly to the condenser's nozzles. Attach the bottom tube to a cold water tap and allow the other tube to go down the drain. Start the cold water flowing before the addition of the volatile solvent.

Plug in the heating mantle and adjust the cooling water to ensure that the condenser stays cool enough to condense the solvent below its midpoint. As the solvent comes to a rolling boil, unplug the mantle. *It will stay hot for quite some time!* The flask will also be hot. With the “condensation ring” still visible in the condenser, measure the boiling temperature. Record the current atmospheric pressure, since it is equal to the vapor pressure above the boiling mixture. The thermometer should be wet; in other words, vapor should condense on it during the experiment. (But don't immerse it.)

Add each solute *aliquot* (means *a measured amount*) from the 5-ml pipette by dropping it straight down the condenser tube. The boiling may stop simply because the solute is at room temperature, but it will most likely resume as the heat from the (still unplugged) mantle continues warming the mixture. If boiling does not resume with sufficient vigor to make a condensation ring obvious, plug in the mantle briefly. If the condensation ring becomes too high, turn on more cold water. *Be careful* when you do this -- the tubing might pop off the condenser! As a last resort if the condensation ring threatens to rise out of the condenser, raise the glassware up out of the mantle by sliding its clamp.

Allow the temperature reading to come to its equilibrium after each addition to obtain an accurate reading of the mixture's boiling point. Do not add so much solute that the boiling point approaches the thermometer's maximum temperature!

At the end of the experiment, turn off and remove the heating mantle (*be careful*, it's hot!) to permit the flask and mixture to cool to a comfortable temperature. **Discard these reagents in the non-halogenated organic waste bottle.** Also turn off the water, drain the condenser into the sink, and clean and return all borrowed equipment. Be sure the heating mantle has cooled to room temperature.

Mixture 2

$V_{(\text{hexane})}$ ml	$V_{(\text{isopropanol})}$ ml	$m_{(\text{hexane})}$	$m_{(\text{isopropanol})}$	$n_{(\text{hexane})}$	$n_{(\text{isopropanol})}$	$X_{(\text{hexane})}$	$X_{(\text{isopropanol})}$
50	0.00						
50	5						
50	10						
50	15						
50	20						
50	25						
50	30						
50	35						
50	40						
50	45						
50	50						

$X_{(\text{Hexane})}$	T	$P^{\circ}_{(\text{hexane})}$	$P^{\circ}_{(\text{isopropanol})}$	P_T

Discussion

Experiment No (4)

Ionization and Electrolyte Behavior

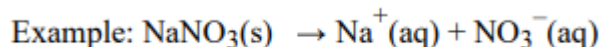
Objects: In this experiment, you will

- Use a Conductivity Probe to measure the conductivity of solutions.
- Investigate the conductivity of solutions resulting from compounds that dissociate to produce different numbers of ions.

Theory: The size of the conductivity value depends on the ability of the aqueous solution to conduct electricity. Strong electrolytes produce large numbers of ions, which results in high conductivity values. Weak electrolytes result in low conductivity, and non-electrolytes should result in no conductivity. In this experiment, you will observe several factors that determine whether or not a solution conducts, and if so, the relative magnitude of the conductivity. Thus, this simple experiment allows you to learn a great deal about different compounds and their resulting solutions. Keep in mind that you will be encountering three types of compounds and aqueous solutions:

- Ionic Compounds:

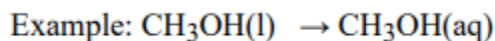
These are usually strong electrolytes and can be expected to 100% dissociate in aqueous solution.



- Molecular Compounds:

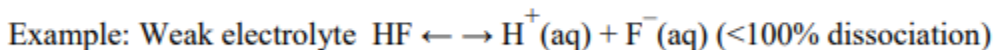
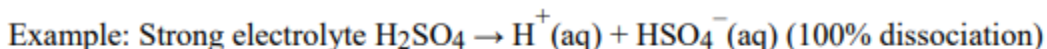
These are usually non-electrolytes. They do not dissociate to form ions. Resulting solutions do

not conduct electricity.



- Molecular Acids

These molecules can partially or wholly dissociate, depending on their strength.



Chemical and apparatus:

A list of substances to be tested is given on the report sheet. Your instructor may do this experiment as a demonstration or individual conductivity meters may be given.

Procedure:

1. In a clean-dry beaker, test the conductivity of different substances and solutions by following the table in your report using the conductivity meter.
2. Answer the questions below the table.

Report of experiment (4)

Students' Names:

Object:

Solution	Conductivity ($\mu\text{S}/\text{cm}$)
H ₂ O distilled	
Glucose (1M)	
NaCl (1M)	
NaCl (2M)	
NaCl(1M) at 60 C°	
H ₂ SO ₄ (1M)	

Answer the following questions:

1. Why did the conductivity increase in (2M) NaCl solution than (1M) NaCl solution?
2. Why does glucose solution consider as a non-electrolytes solution?
3. When heat was applied in (1M) NaCl solution, why the conductivity was increased?
4. Why H₂SO₄ solution is the strongest electrolytes in the list that was given?

Experiment No (5)

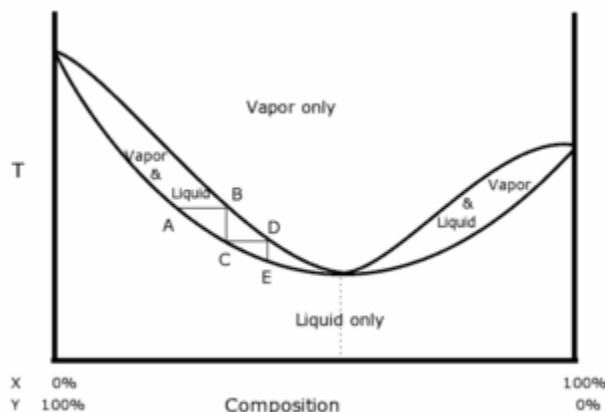
Temperature-Composition Diagram of a Binary Mixture

Objects:

The aim of this experiment is to:

1. Study and draw the temperature-composition diagram for ethanol- water solution.
2. Determine the temperature and composition of the azeotrope.

Theory: Distillation is one of the most useful methods for the separation and purification of liquids. There are many types of distillation such as simple distillation, fractional distillation, and steam distillation. The difference in boiling points between the compounds is what determines what type should be used. In the case of non-ideal solutions, it might not be possible to separate compounds completely. The systems that are impossible to separate completely are known as azeotropes (azeotrope = not changing composition when boiling). For example about this case is the separation of ethanol-water solution.

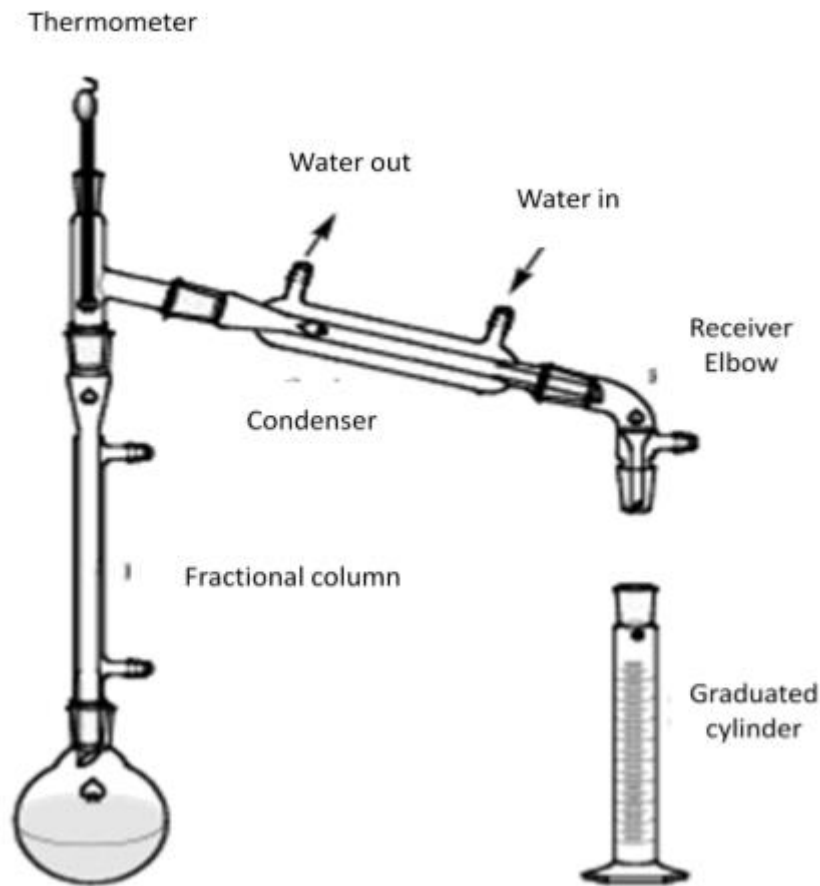


Chemicals and Apparatus

Distillation Setup: Thermometer, 250-ml round bottom flask, fractional column, condenser, receiver Elbow, 250 mL- graduated cylinder, Flask heater, rubber adapter, grains of pumice, mantle, ethanol, distilled water.

Procedure

1. Prepare ethanol solutions with 40, 60, 80, and 95%. Of concentration.
2. Setup the distillation apparatus as showing below :



3. In the 250-ml round bottom flask add 100 ml of 40% ethanol solution and some grains of pumice.
4. Turn on the mantle hot plate (do not apply high temperature).
5. The solution will start to boil and evaporate and then the resulting steam will condense and fall in the graduated cylinder.
6. When the first drop falls, record the boiling and the evaporation temperature in the results table.
7. Repeat the same steps for other solutions.

Calculation

1. Plot T versus x (W %) using your results.
2. Draw smooth curves through the points obtained from boiling temperatures and through the points obtained from evaporation temperatures.
3. Label all the phases present on the graph.
4. Determine the azeotropic composition and its boiling point from your graph.

Report

Students' Names:

Object:

W %	T_b (boiling temperatures)	T_v (evaporation temperatures)
0		
40		
60		
80		
95		
99		
100		

• From the graph answer the following questions :

1- In what concentration does the azeotropic occur?

2- What is the boiling point for the azeotropic ?

Experiment No (6)

phase diagram for two partially miscible liquids

Object: to construct a phase diagram of two immiscible liquids (aniline-heptane system) and find out the critical solution temperature of this system.

Theory: When two partially miscible liquids are mixed and shaken together, we get two solutions of different composition. For example, on shaking aniline and heptane, we get two layers; the upper layer is a solution of heptane in aniline and the lower layer is a solution of aniline in heptane. At a fixed temperature, the composition of each solution is fixed and both the solutions are in equilibrium. The composition of the two layers (phases) at equilibrium varies with the temperature. Raising the temperature increasing the miscibility of the two solutions. Above a particular temperature, such solutions are completely miscible in all proportions. This temperature is known as **Upper Critical Solution Temperature**. Figure (1) shows the temperature-composition diagram for a mixture of A and B. The region below the curve corresponds to the compositions and temperatures at which the liquids are partially miscible. The upper critical temperature T_c is the highest temperature at which phase separation occurs and above T_c the two solutions are fully miscible.

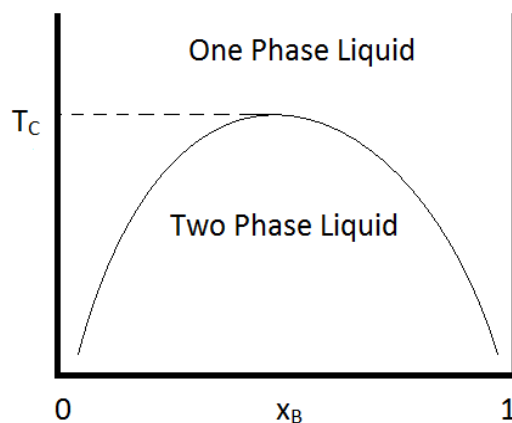


Figure 3 The temperature composition diagram of a mixture of A and B.

Chemical and Apparatus: Aniline, heptane, water bath, thermometer, beaker and pipette.

Procedure:

1. 10 ml of aniline was measured into a test tube and then 1 ml of heptane added to it.
2. The two-phase solution was then heated in a water bath until a single-phase solution was formed.
3. The temperature at which the single-phase formed was measured and recorded.
4. The volume of aniline was kept varying with respect to heptane as shown in the below table.
5. A curve is plotted with temperature as ordinate against mass fraction of aniline as abscissa. The maxima of the solubility curve will give the critical solution temperature.

Density of heptane = 0.706 g/ml, density of aniline = 1.023 g/ml.

Mass = density \times volume.

$$\text{Mass fraction} = \frac{\text{mass of aniline}}{\text{mass of aniline} + \text{mass of heptane}}$$

Precautions; Aniline is toxic and can be absorbed by the skin, care must be taken not to spill.

Report

Students' Names:

Object:

Table of Results:

Vol. of aniline	Vol. of heptane	Temperature	Mass frac. of aniline.
10	1		
9	2		
8	3		
7	4		
6	5		
5	6		
4	7		
3	8		
2	9		
1	10		

Discussion:

Experiment No (7)

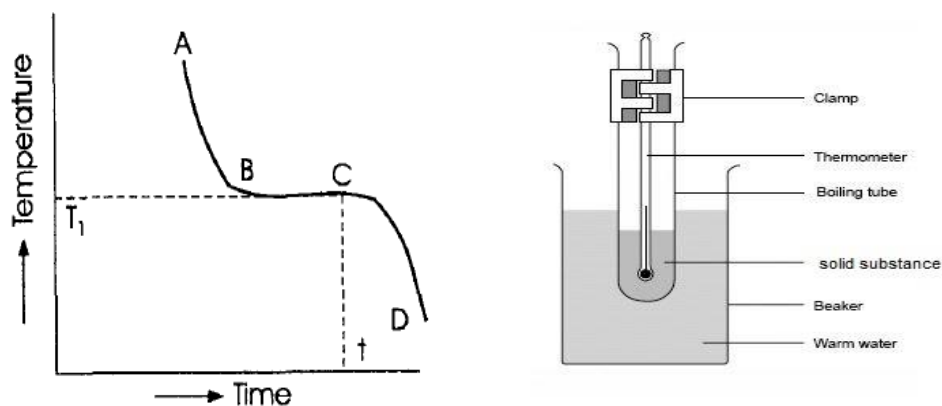
Cooling curves for Pure substances

Object:

- 1- To draw cooling curves for pure substances (Naphthalene, α -Naphthol) and find their melting points from the curve.

Theory:

A cooling curve for a substance is a curve between temperature and time of cooling. It is started when the entire mass is liquid and is continued till it is completely solidified. The curve will be continuous, as long as there is no transition or change of phase. At a certain temperature, the liquid begins to solidify and heat is found to be liberated during this phase change of liquid to solid. This method is known as **thermal analysis**. Due to this liberation of heat the cooling curve (Fig.1) becomes discontinuous. The cooling curve ABCD consists of three parts.



1. AB, which corresponds to the cooling of the liquid. At temperature T_1 , the liquid begins to solidify.
2. BC, which corresponds to the separation of the solid. The solid is formed at a rate just sufficient to counterbalance the heat loss by radiation and hence the temperature remains constant. At time t , the liquid is completely converted into solid. This steady temperature is the melting point of the substance.
3. CD, which corresponds to the cooling of the solid phase of the substance.

Note; The melting or freezing temperature is the temperature at which the solid and liquid phases of a substance can exist in equilibrium. Like any equilibrium state, the chemical potential of a substance is the same throughout a sample. Regardless of how many phases are present.

Chemicals and Apparatus

Naphthalene, α -Naphthol, Boiling tube, 1-L beaker, Thermometer (0-100°C), hot plate, Stop watch, Clamp, stand and boss.

Procedure

1. Put about 800 ml water into the beaker and heat it on the hot plate until the water starts to boil.
2. In a boiling tube put 1 g of naphthalene, fit a thermometer in this tube then place it in a boiling water bath, as shown in Fig 2.
3. Shake gently with the thermometer until the substance melts completely.
4. Remove The beaker from the hot plate, keep the tube in the beaker and record the temperature every 30 sec, (If the rate of cooling is slow, observe reading after a minute) until it becomes constant and the whole mass solidifies (note at which temperature you see the solid start to melt).
5. Plot the cooling curve between temperature (ordinate) and time (abscissa) and note the melting temperature of naphthalene which is characterized by the temperature of the plateau (stability of temperature with time).
6. Repeat experiment with 1 g of α -Naphthol and determine its melting temperature.

Experiment No (8)

Determination of the binary solid-liquid diagram for (Naphthalene and α -Naphthol)

Object: To draw a phase diagram for Naphthalene and α -Naphthol and from it find the eutectic temperature of the binary system.

Theory: If a mixture of two components A and B (melting point of A is higher than that of B) is melted and cooled and if the percentage of A is higher than that of B in the mixture, then it is seen that the melting point of A will be lowered due to the presence of B. At a certain temperature, known as **Transition Temperature**, solid A will crystallize earlier, leaving the liquid richer in B. After some more cooling, the composition of the liquid will reach a ratio of A to B so as to allow the separation of A and B simultaneously in the crystalline form. Thus, that temperature at which the two solids A and B are in equilibrium with the solution is known as **Eutectic Temperature** (Greek: eutectic = easy melting).

Figure (1) depicts the cooling curve of a mixture of two components A and B (Notice; the shape is not like a cooling curve of pure substance). The first break in the curve occurs at B_1 where one of the components, begins to solidify. During this solidification process, heat is liberated, and the temperature falls more slowly. It is to be noted that the temperature does not remain constant because the composition of the mixture (amount of A: amount of B) is changing continuously whereby the temperature of solidification correspondingly changes. At a certain temperature T_2 , known as **Eutectic Temperature**, the remaining liquid solidifies, both the components A and B crystallize out simultaneously.

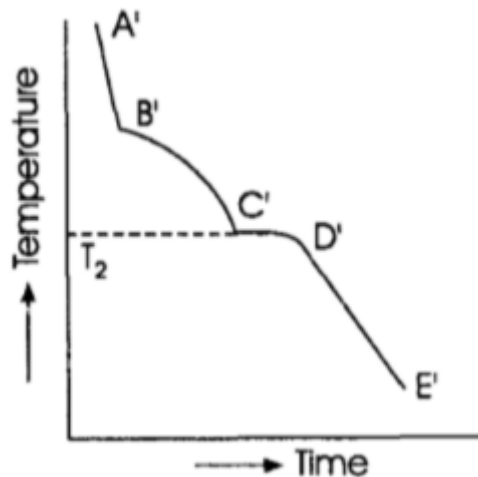
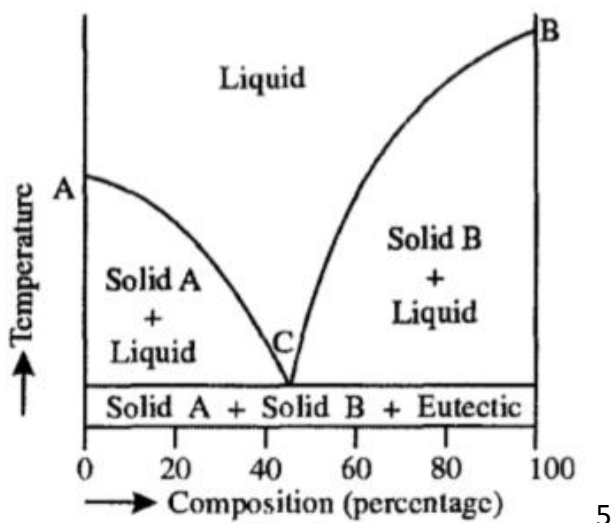


Figure 4

If we draw cooling curves for several mixtures of A and B and plot the corresponding **Transition Temperatures** as ordinate against composition (Mole fraction) as abscissa,

we get a phase diagram [fig. (2)], from which we can find the melting points of the two components as well as the **Eutectic Temperature** of the system.



Chemicals and Apparatus

Naphthalene, α -Naphthol, Boiling tube, 1-L beaker, Thermometer (0-100°C), hot plate, stopwatch, Clamp, stand and boss.

Procedure

1. Put about 800 ml water into the beaker and heat it on the hot plate until the water starts to boil.
2. Prepare the following mixtures of Naphthalene and α -Naphthol by weighing them separately.

Mixture	1	2	3	4	5
Naphthalene	0.2 g	0.4 g	0.5 g	0.7 g	0.9 g
α -Naphthol	0.8 g	0.6 g	0.5 g	0.3 g	0.1 g

3. In a boiling tube put the first mixture, fit a thermometer in this tube then place it in a boiling water bath. Shake gently with the thermometer until the mixture melts completely.
4. Remove The beaker from the hot plate, keep the tube in the beaker and record the temperature every 30 sec, (If the rate of cooling is slow, observe reading after a minute), until it becomes constant and the whole mass solidifies. (note at which temperature you see the solid start to melt).

5. Remove the thermometer and pour out the contents of the tube. Wash and dry it and repeat the above process for the remaining mixture of Naphthalene and α -Naphthol.

- 1) Draw a cooling curve for every mixture of Naphthalene and α -Naphthol (time is taken as abscissa while temperature as ordinate). From the cooling curves, find the transition temperature for each mixture and tabulate the results as follows:

Mixture	Mole fraction of Naphthalene	Transition temperature
1 (0.2: 0.8)		
2 (0.4: 0.6)		
3 (0.5: 0.5)		
4 (0.7: 0.3)		
5 (0.9:0.1)		

- 2) Draw a curve with transition temperature as ordinate and mole fraction of naphthalene as abscissa and indicate on this solid-liquid diagram the legend of different zones (Liquid phase, solid phase, Eutectic point).