336 CHEM

PHYSICAL CHEMISTRY OF SOLUTIONS (Laboratory)

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# **Laboratory Safety and Guidelines:**

1. Approved safety goggles or eye shields must be worn at all times to guard against

the laboratory accidents of others as well as your own. Contact lenses should be

replaced with prescription glasses. Where contact lenses must be worn, eye

protection (safety goggles) is absolutely necessary. A person wearing prescription

glasses must also wear safety goggles or an eye shield.

1. Shoes must be worn. Wear only shoes that shed liquids. High-heeled shoes; open toed shoes; sandals; shoe tops of canvas, leather, or fabric straps or other woven material are not permitted
2. Clothing should be only non-synthetic (cotton). Shirts and blouses should not be torn, frilled, frayed, or flared. Sleeves should be close-fit. Clothing should cover the skin from “neck to below the knee (preferable to the ankle) and at least to the wrist.” Long pants that cover the tops of the shoes are preferred
3. Laboratory aprons or coats (nonflammable, nonporous, and with snap fasteners) are highly recommended to protect outer clothing
4. Gloves are to be worn to protect the hand when transferring corrosive liquids. If you are known to be allergic to latex gloves, consult with your instructor.
5. Jewelry should be removed. Chemicals can cause a severe irritation if concentrated, under a ring, wristwatch, or bracelet; chemicals on fingers or gloves can cause irritation around earrings, necklaces, and so on. It is just a good practice of laboratory safety to remove jewelry.
6. Secure long hair and remove (or secure) neckties and scarves.
7. Never taste, smell, or touch a chemical or solution. Individual allergic or sensitivity responses to chemicals cannot be anticipated. Poisonous substances are not always labeled.
8. Wash your hands often during the laboratory, but always wash your hands with soap and water before leaving the laboratory! Thereafter, wash your hands and face in the washroom. Toxic or otherwise dangerous chemicals may be inadvertently transferred to the skin and from the skin to the mouth.
9. **Report all accidents or injuries**, even if considered minor, immediately to your instructor.
10. If **an accident occurs**, do not panic! The most important first action after an accident is the care of the individual. Alert your laboratory instructor immediately! If a person is injured, provide or seek aid immediately. Clothing and books can be replaced, and experiments can be performed again later. Second, take the appropriate action regarding the accident: clean up the chemical, use the fire extinguisher, and so on.
11. Whenever your skin (hands, arms, face, etc.) comes into contact with chemicals, quickly flush the affected area for several minutes with tap water followed by thorough washing with soap and water. Use the eyewash fountain to flush chemicals from the eyes and face. Get help immediately. Do not rub the affected area, especially the face or eyes, with your hands before washing.
12. Treat chemical spills in the laboratory as follows:
13. Alert your neighbors and the laboratory instructor
14. Clean up the spill as directed by the laboratory instructor
15. If the substance is volatile, flammable, or toxic, warn every one of the accidents.

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**The following rules must be followed:**

1. Smoking, drinking, eating, and chewing are not permitted at any time because chemicals may inadvertently enter the mouth or lungs. Your hands may be contaminated with an “unsafe” chemical. Do not place any objects, including pens or pencils, in your mouth during or after the laboratory period. These objects may have picked up a contaminant from the laboratory bench.
2. Maintain an orderly, clean laboratory desk and drawer. Immediately clean up all chemical spills, paper scraps, and glassware. Discard wastes as directed by your laboratory instructor.
3. Be aware of your neighbors’ activities: You may be a victim of their mistakes. Advise them of improper techniques or unsafe practices. If necessary, tell the instructor.
4. At the end of the laboratory period, completely clear the lab bench of equipment, clean it with a damp sponge or paper towel (and properly discard), and clean the sinks of all debris. Also clean all glassware used in the experiment.

من رايي ان توضع صور للاحتياطات افضل من الكلام

# **Calculations and Presentation of Data**

## **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.

# **تضاف هنا توزيع الدرجات والميد الفاينل**

# **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** **i**: The change in molar volume V when one mole of Ai is added to an “infinitely large” sample at constant T and P.

 (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, nA 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

VT = nAA + nBB  (2)

It is convenient to express this on a per-mole basis, dividing through by the total number

of moles (nA ‏+ nB) to obtain the equivalent equation in terms of mole fraction XB (with XA = 1 – XB)

 = XAA + XBB = (1- XBA + XBB = A + XBB  - A ) (3)

As shown by the final equation on the right, the equation for = (XB) is that of a straight with slope

 A (4)

And intercept A. The graph below shows the plotted behavior of V(XB) (solid line) from A0 (at XB = 0) to B0 (at XB =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 A (on the left) and B (on the right). The extrapolated-tangent construction therefore makes it easy to read off the partial molar volumes A, B at any desired concentration XB.



**Figure 1**

# **Chemicals and Apparatus**

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

# **Procedure**

1. Prepare a series of solutions by weight containing ethanol and water 20, 40, 60, 80-wt% of ethanol.
2. Determine the density of each solution at room temperature using the following procedure:
	* + - Determine the weight of previously oven-dried and cooled pycnometer.
			- 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. When doing this, make sure that the outside of the pycnometer is completely dry.
			- Given the density of water at ambient temperature, ρ = 0.997 g/ml calculate the volume of pycnometer using equation 5.

V(Pyc) = (5)

* + - * Subsequently weigh the pycnometer, which is filled with different mixtures of ethanol and water then calculate the densities of these solutions.
			* تكتب الطريقه بالتفصيل وتعريف الرموز مثلا كالتالي
			* للتاكد من حجم البيكنوميتر
			* 1- وزن البيكنوميتر فارغ (م1)
			* 2- وزن البيكنوميتر بعد تعبئته بالماء ولتكن (م2)
			* احسبي حجم البيكنوميتر باستخدام القانون التالي ....
			* بعد تحديد حجم البيكنوميتر (ويوضع رمز الحجم ) يتم حساب كثافه المحاليل السابقه كالتالي
			* 1- وزن البيكنوميتر فارغ (م1)
			* 2- ورن البيكنوميتر بعد ملئه بالمحلول (م2)
			* 3- تطبيق المعادله رقم .. وحساب الكثاف
			* 4- توضع النتائج في جدول ثم ترسم العلاقه بين كذا (المحور السيني ) وكذا (المحور الصادي) حيث الميل هو والجزء المقطوع هو
			* 5- يتم رسم العلاقه بين وبين لحساب
			* 6- يتم احساب الحجم المولي عن طريق قسمه الحجم النوعي على ..
			* في التقرير
			* يكتب اسم الطالبه
			* الهدف من التجربه (فراغ هي تكمله )
			* حجم البيكنوميتر (والطالبه تضع القيمه بعد طرح الاوزان على الكثافه )
			* تضعين لها جدول تكمله ثم تطلبين منها إيجاد الحجم المولي فقط
			* هي سوف تتبع إجراءات العملي حيث سوف ترسم العلاقه الأولى ثم الثانيه لايجاد المطلوب \

العملي والحسابات مع بعض لاتضعين عنوانين منفصلين

ويعمل بجميع التجارب كالتالي

لانريد كتابه الكتاب في الملزمه نريد إعادة صياغه بشكل مبسط وواضح وبخطوات تسلسليه

لذا لابد من إعادة الصياغه

# **Calculation**

1. 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.
2. Draw a smooth curve through all points.
3. 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.
4. Calculate the partial molar volume of each component using equation 6.

 = Mwt d-1 (6)

Mwt of ethanol = 46.07 g/mol and Mwt of water = 18.015 g/mol

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

# **Report**

Name:

Q1:

|  |  |  |
| --- | --- | --- |
| m 1= | m 2 = | V (pyc) = |

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| W% | m = m2 – m1 | d-1  |  (EtOH) |  (water) | VT |
| 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

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# **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

PA = P°A XA (1)

Similarly, for the other component B,

PB = P°B XB (2)

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

PT = P°A XA  + P°B XB (3)

When a solution is boiling, its vapor pressure (PT) 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.

) (4)

P Vap =

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 Tbp is pure liquid’s normal boiling point. 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!



 Warm water out →

 ←Cold water in

 30°C and beyond visible→

**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)

1. Using the Clausius-Clapeyron equation (2) to calculate the vapor pressure P° above each pure liquid for each measured T.
2. 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°A XA + P°B XB will be the mixture’s vapor pressure, the current atmospheric pressure). In other words, to what extent P°A XA + P°B XB is equal to unit

**hint 1: Use the following physical data**

|  |  |  |  |
| --- | --- | --- | --- |
| **Reagent** | **T°bp °C** | **Density ( g/ml)** | **Δ Hvap at 25°C, kJ/mol** |
| Pentane | 36.06 |  | 26.4 |
| Heptane | 98.5 |  | 36.57 |
| Isopropanol | 82.3 |  | 45.39 |
| Hexane |  |  |  |

**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.

# **Report**

**Report hints:** include your data and graphs in your Results section. In the Discussion, describe the influence of any non-idealities on your results. Is Raoult's Law obeyed? Why or why not? Determine the extent to which you might ignore the vapor pressure of the solutes in both experiments; in other words, what would the solute vapor pressures be near the solvent's boiling point? Are they negligible? Use the Clausius-Clapeyron equation in your answer.

# **Results**

## **Mixture 1**

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| **V(Hexane) ml** | **V(Heptane) ml** | **m(Hexane)** | **m(Heptane)** | **n(Hexane)** | **n(Heptane)** | **X(Hexane)** | **X(Heptane)** |
| 50  | 0.00 |  |  |  |  |  |  |
| 50 | 5 |  |  |  |  |  |  |
| 50 | 10 |  |  |  |  |  |  |
| 50 | 15 |  |  |  |  |  |  |
| 50 | 20 |  |  |  |  |  |  |
| 50 | 25 |  |  |  |  |  |  |
| 50 | 30 |  |  |  |  |  |  |
| 50 | 35 |  |  |  |  |  |  |
| 50 | 40 |  |  |  |  |  |  |
| 50 | 45 |  |  |  |  |  |  |
| 50 | 50 |  |  |  |  |  |  |

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| --- | --- | --- | --- | --- |
| **X(Hexane)** | **T** | **P°(hexane)** | **P°(Heptane)** | **PT** |
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## **Mixture 2**

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| **V(hexane) ml** | **V(isopropanol) ml** | **m(hexane)** | **m(isopropanol)** | **n(hexane)** | **n(isopropanol)** | **X(hexane)** | **X(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(Hexane)** | **T** | **P°(hexane)** | **P°( isopropanol)** | **PT** |
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# **Discussion**

# **Experiment No (4)**

**Determination of molar mass of pure substance by osmometry** **method**

**Object:** The aim of this experiment is to determine the molar mass of an unknown compound by using the osmotic pressure feature.

**Theory:** Osmosis is the movement of water from an area of low concentration of solute to an area of higher concentration of solute. A solute is atoms, ions, or molecules dissolved in a liquid. The rate of osmosis is determined by the total number of particles dissolved in the solution. The more particles dissolved, the faster the rate of osmosis. However, water movement applies an osmotic pressure that can stop the osmosis process.

 osmotic pressure ( ) can be calculated by using the following equation:

 Where d is the density of the solution, g is the gravitational acceleration (= 9.81 m s-2), and h is height of solution.

In diluted solutions:

Where are:

π: osmotic pressure. C: strength of solution(g/L)

R: universal gas constant. T: temperature(K)

MWt: the molar mass of unknown compound.

# **Chemicals and Apparatus**

Unknown compound that is soluble in water, distillated water, 6. membranes (commercial cellophane film), 500- mL beaker ,thistle tube, rule thermometer, and elastic tape.

**Procedure:**

1. Prepare 0.5, 1, 1.5, and 2 (g/L) solutions.

2. Calculate the density of each solution.

3. Prepare your osmometer tube:

4. Closes the tube from its largest diameter side with the membrane film by securing it with an elastic tape.

5. Filled to half of the tube with the prepared solution.

6. - Note the height value of the liquid in the tube using a simple rule.

7. Put the tube containing the solution into a 500 mL-beaker containing about 250 mL of distillated water and fixe it using a stand.

8. Wait for the equilibrium to be realized between the solution inside the tube and solvent (water) in the beaker, then note the new height of the liquid in the tube.

9. Repeat this procedure for different concentrations of the solution and records the values of their height in the previous Table.

**Calculation:**

Calculate the density of each solution.

Calculate the osmotic pressure ( ) for each solution.

 Plot versus c .

Find out the slope which equal to ()

Calculate the molar mass of unknown compound.

**Report**

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **C (g/L)** | **0.5** | **1** | **2** | **3** |
| **h(m)** |  |  |  |  |
| **d(kg/m3)** |  |  |  |  |
| **π (kg/m.s2)** |  |  |  |  |

**Plot versus c**

**Slope =**

The molar mass (MWt) of unknown compound =

# **Experiment No (6)**

**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, distillated water.

# **Procedure**

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



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

|  |  |  |
| --- | --- | --- |
| **W %** | **Tb**(boiling temperatures) | **Tv**(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 (7)**

## **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 Tc is the highest temperature at which phase separation occurs and above Tc 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 × volume.

Mass fraction =

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

**Report**

**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 (8)**

# **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 T1, 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.

# **Report**

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Time  | Temperature |  | Time | Temperature |
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**Plot the variation of the temperature as a function of time and determine the following:**

Melting point of Naphthalene =

Melting point of α-Naphthol =

**Q) Why the temperature remains constant during a transformation from one state to another state?**

# **Experiment No (9)**

**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 B1 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 T2, known as **Eutectic Temperature**, the remaining liquid solidifies, both the components A and B crystallize out simultaneously.



Figure

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, 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. 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 | * 1. g
 | * 1. g
 |

1. 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.
2. 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).
3. 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.

# **Report**

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Mixture 1 | Mixture 2 | Mixture 3 | Mixture 4 | Mixture 5 |
| Temp | Time | Temp | Time | Temp | Time | Temp | Time | Temp | Time |
|  |  |  |  |  |  |  |  |  |  |
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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) |  |  |

1. 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).

# **Experiment No (5)**

**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 (9)**

|  |  |
| --- | --- |
| **Solution** | **Conductivity (µS/cm)** |
| H2O distilled |  |
| Glucose (1M) |  |
| NaCl (1M) |  |
| NaCl (2M) |  |
| NaCl(1M) at 60 C° |  |
| H2SO4 (1M) |  |

**Answer the following questions:**

1. Why did the conductivity increases in (2M) NaCl solution than (1M) NaCl solution?
2. Why dose 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?