Experiment 2

Distillation-Fractional Distillation

Distillation-Fractional Distillation:

Distillation is the process of heating a **mixture** to the point that it boils – that is, the vapor pressure of the liquid equals the atmospheric pressure, and vapor will start forming below the surface of the liquid.

If the resulting vapor is condensed, the condensed liquid will often contain more of the volatile component of the mixture than others. This allows **purification** by distillation.

Distillation is a technique that is used to

- ❖obtain a boiling point of a pure liquid.
- ❖purify a mixture of liquids based on boiling point differences among compounds.

It has wide applications in petroleum refineries and the chemical industry.

In practice, separation of a liquid mixture into its components by a single distillation (simple distillation) is possible *when the boiling points of the components are 70-80 degrees or more apart.*

2

Fractional Distillation

For mixtures of liquids having boiling points much **less than 80** degrees apart, separation can be achieved only by **fractional distillation**.

To achieve complete separation, the liquid mixture may have to be **vaporized and condensed many times**. This is best accomplished by using a fractionating column. (Fractional distillation).

The fractionating column provides a **large surface area for continuous heat exchange** between the hot ascending vapor and the cooler descending liquid, thus resulting in a series of evaporations and condensations leading to separation of the two components.

Each step makes the vapor richer and richer in the more volatile component.

3

Theoretical Plates and Fractional Distillation:

The term theoretical plates is used to express how many times the liquid is vaporized and condensed in the column. **HETP** refers to the height of the column equivalent to one theoretical plate.

The behavior of a solution of two miscible liquids, A and B, is best explained by referring to **Raoult's law** which states that

The partial pressure of liquid A (p_A) in a mixture is equal to the vapor pressure of pure liquid A (P°_A) multiplied by the mole fraction of A in the mixture (X_A).

The same applies to liquid B. Therefore:

$P_A = X_A . P^\circ_A$ and $P_B = X_B . P^\circ_B$

From **Dalton's law**, the total vapor pressure of the solution (P_T) is the sum of the partial pressures of A and B:

$P_T = p_A + p_B = X_A.P^\circ_A + X_B . P^\circ_B$ $(X_A + X_B = 1)$

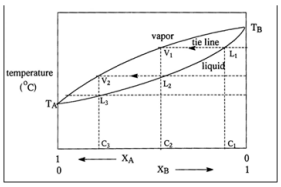
4

Vapor-Liquid Composition for an Ideal Mixture of Two Liquids

A solution of A and B will boil when the total vapor pressure **(P_T)** equals the **external pressure**. This occurs at a temperature which is *intermediate* between the boiling points of the two pure liquids (lower curve in the below Figure).

This diagram shows the temperature at which mixtures of A and B of various compositions boil (lower curve).

The **composition** of the vapor in equilibrium with the liquid is given by the tie line connecting the liquid and vapor curves. It is clear from the Figure that the vapor will always be richer than the liquid in the more volatile components. This makes sense, since the molecules of the more volatile component will escape more readily, and thus be in higher proportion in the vapor phase.



5

Azeotropes:

Unfortunately, distillation is not always successful for purifying organic liquids. Some liquids form constant boiling mixtures called **azeotropes**.

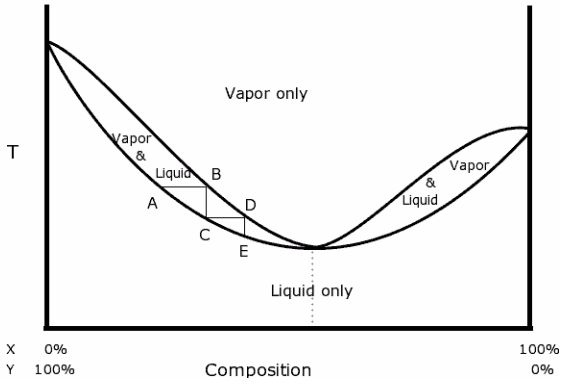
An azeotropic mixture has a fixed composition in the liquid phase and the same fixed composition in the vapor phase. This liquid will have a fixed boiling point and will act a pure compound.

Its proportions cannot be altered by simple distillation. This happens because, when an azeotrope is boiled, the vapor has the same proportions of constituents as the unboiled mixture.

Because their composition is unchanged by distillation, **azeotropes** are also called (especially in older texts) **constant boiling mixtures**.(see the Figure on next page)

6

Azeotropes:



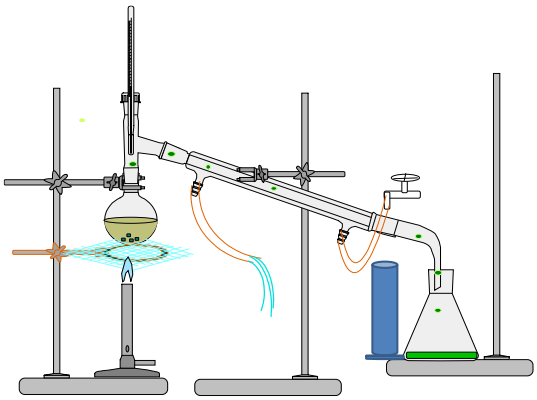
7

Simple distillation setup

Make sure that the boiling is smooth-
No bumping.

Adjust the flame if necessary.

Record the temperature and volume of distillate.



8

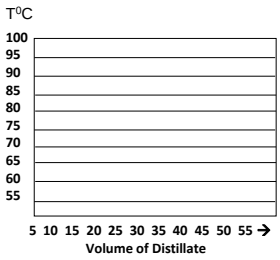
Procedure: Simple Distillation

Make a mixture of the two liquids (acetone-water) 50 mL each and pour it into a 250 mL round bottomed flask.

Add 2-3 boiling chips (**anti-bumping granule**) to prevent the liquid from bumping due to superheating.
Begin the distillation so that one drop per second is collected in the graduated cylinder (receiver) .
Record the temperature at each 5-mL interval.

Do not distill to dryness.
{ At 760 mm Hg, pure acetone boils at 56^{o}C and pure water at 100^{o}C}.

Plot the data in the graph below.



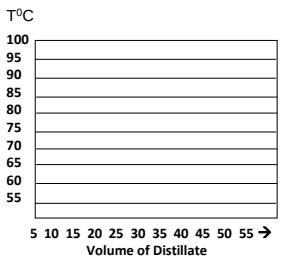
9

Procedure: Fractional Distillation

Make the mixture of the two liquids (acetone-water) 50 mL each and pour it into a 250 mL round bottomed flask (or Combine the two fractions collected from simple distillation).

Add 2-3 boiling chips, attach the fractionating column and proceed as for simple distillation.

Record the temperature at each 5 mL interval and plot the data in the graph below.



10