Distillation

Purpose of experimental

1. Separate a liquid solvent mixture using both simple and fractional distillation.
2. Determine the boiling point of the purified solvent

Theory part of experimental

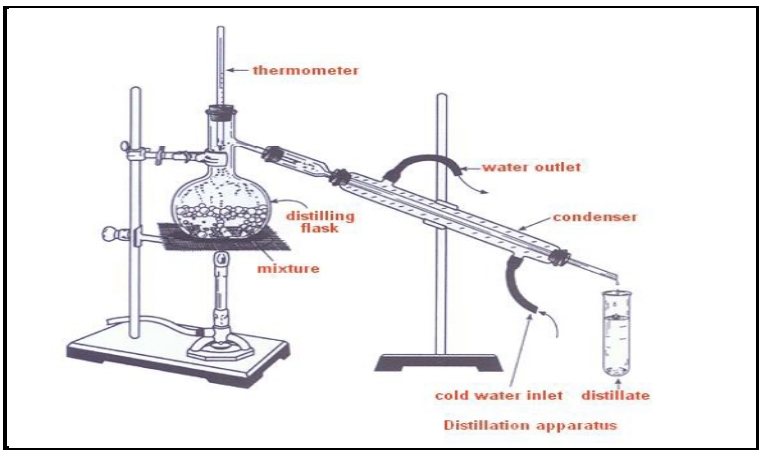
Distillation is the most common method used to separate and purify liquids. **The process consists of** heating a liquid to its boiling point and conducting the vapors to a cooling device where they are condensed. The condensate is then collected. The vapor pressure of a liquid is defined as the pressure that is exerted by a liquid at a given temperature. This force is due to the molecules escaping from the liquid's surface into the gas phase. The vapor pressure of a liquid increases with increasing temperature. The boiling point of a liquid is that temperature at which the vapor pressure is equal to the pressure of the surroundings. If a flask is open to the air, the vapor pressure at the boiling point is equal to the atmospheric pressure. The boiling points of many liquids are listed in the literature referenced to 760 mm Hg (standard pressure).

The boiling point of a liquid is determined by placing a thermometer in the vapor. The temperature of the vapor will remain constant throughout the distillation if the vapor is pure. The boiling point at a given pressure is a characteristic property of the pure compound just as the melting point of a pure crystalline compound. If a mixture of two miscible liquids with different boiling points is heated to boiling, the vapor will not have the same composition as the liquid; it will be richer in the more volatile component. In order to obtain pure components, the now enriched

mixture must be redistilled. A **fractionating column** is a device for increasing the efficiency of the distillation process. It consists of a vertical column packed with inert materials or with indentations which increase the surface area of the column. As the hot vapors of the liquid rise, they condense on the greater surface area. As the condensate flows back down to the boiling flask, the liquid is revaporized as it comes in contact with the hotter portion of the lower column. This is repeated many times, until the final distillate is nearly pure. By using select columns, liquids with boiling points just 2° C apart can be separated. Some liquid mixtures do not form nearly**.**

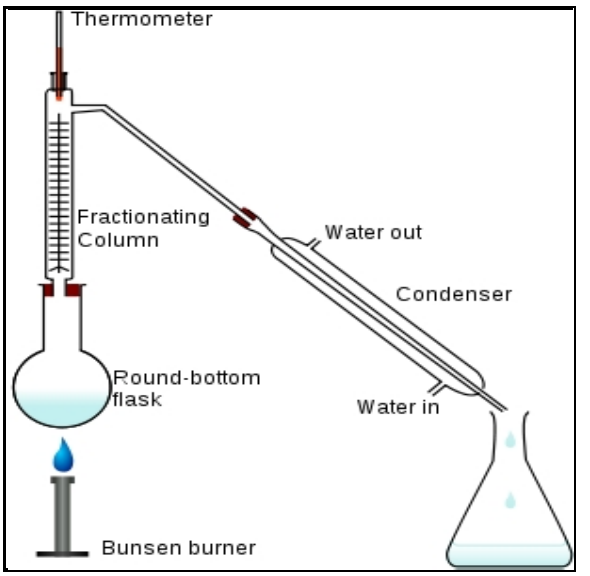
Types of distillation

1- **Simple distillation**: in simple distillation, the vapor is immediately channeled into a condenser. Consequently, the distillate is not pure but rather its composition is identical to the composition of the vapors at the given temperature and pressure. If the difference in boiling points is greater than 25°C, a simple distillation is used.



Simple distillation apparatus

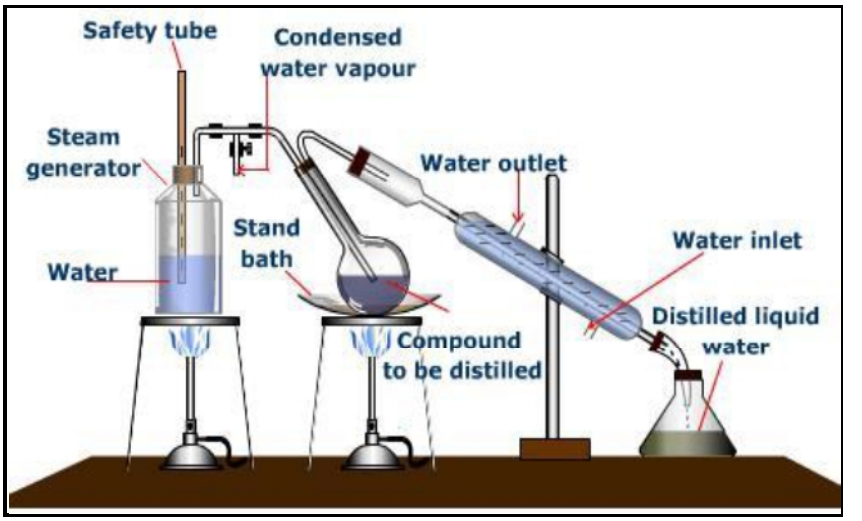
2- Fractional distillation: fractional distillation is the separation of a mixture into its component parts, or fractions, such as in separating chemical compounds by their boiling point by heating them to a temperature at which several fractions of the compound will evaporate. It is a special type of distillation. Generally the component parts boil at less than 25°C from each other under a pressure of one atmosphere (atm).



Fractional distillation apparatus

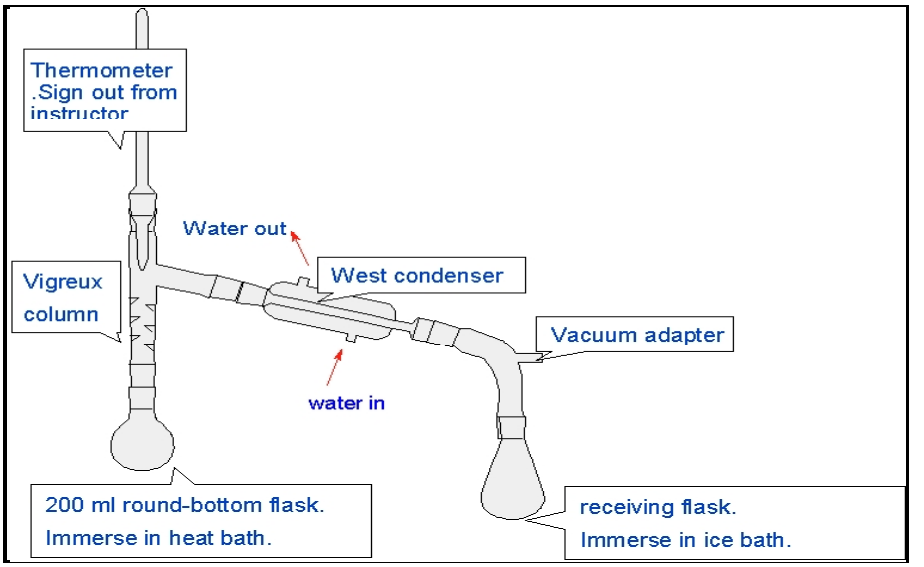
3- Steam distillation: steam distillation is a method for distilling compounds which are heat-sensitive. The temperature of the steam is easier to control than the surface of a heating element, and allows a high rate of heat transfer without heating at a very high temperature. This process involves bubbling steam through a heated mixture of the raw material. The vapor mixture is cooled and condensed, usually yielding a layer of oil and a layer of water.

Steam distillation of various aromatic herbs and flowers can result in two products; an essential oil as well as a watery herbal distillate.



Steam distillation apparatus

4- Vacuum distillation: vacuum distillation Some compounds have very high boiling points. To boil such compounds, it is often better to lower the pressure at which such compounds are boiled instead of increasing the temperature. Once the pressure is lowered to the vapor pressure of the compound (at the given temperature). This technique is also very useful for compounds which boil beyond their decomposition temperature at atmospheric pressure and which would therefore be decomposed by any attempt to boil them under atmospheric pressure.



Vacuum distillation apparatus

Pieces of distillation apparatus

- heat source, such as a hot plate with a bath
- distilling flask, typically a round-bottom flask
- receiving flask, often also a round-bottom flask
- fractionating column (Vigreux column)
- distillation head
- thermometer
- condenser
- boiling chips

Boiling chips: are small, insoluble, porous **stones** made of calcium carbonate or silicon carbide. These **stones** have pores inside which provide cavities both to trap air and to provide spaces where bubbles of solvent vapor can form.

Experimental No. (8) Distillation

Application of distillation

The application of distillation can roughly be divided in four groups: laboratory scale, industrial distillation, distillation of herbs for perfumery and medicinals (herbal distillate), and food processing.

Commercially, distillation has a number of uses. It is used to separate crude oil into more fractions for specific uses such as transport, power generation and heating. Water is distilled to remove impurities, such as salt from seawater.

Chemical and apparatus

$KMnO_4$ solution, boiling chips, thermometer, distillation head, hot plate , distilling flask (typically a round-bottom flask),receiving flask (often also a round-bottom flask).

Procedure of experimental (simple distillation)

1- Place 50 mL your unknown mixture ($KMnO_4$ solution) with boiling chips in a 100 mL round-bottom flask equipped with a hot plate
2- Distill the mixture slowly to obtain the best result, increase the temperature very gradually.
3- Even though your heating mantle is heating, the temperature that the thermometer displays doesn't change much until after the liquid boils. Why?
4- As the distillation proceeds, record the temperature and the total volume of the distillate collected in your laboratory notebook.

Calculation

$$\% \text{ Yield} = \frac{\text{Volume of product}}{\text{Volume of sample}} \times 100$$

Questions for discussion

1. What is the purpose of the boiling chips?
2. In this lab you will separate a mixture of organic liquids by two methods; simple and fractional distillation. In theory, which method should give the better separation? Briefly explain why?
3. What effect is produced on the boiling point of a solution by a soluble, non-volatile substance? What is the effect produced on the boiling point of a liquid by an insoluble substance? What is the vapor temperature above the surfaces of these to liquids?
4. Why doesn't a pure liquid distill all at once when the boiling point is reached?
5. Why is a packed column more efficient than an unpacked column for fractional distillation?
6. Why does slow distillation result in better separation of two liquids than fast distillation?
7. Why is it dangerous to attempt a distillation in a completely closed system?