Distillation

Introduction

Since organic compounds do not usually occur in pure condition in nature, and are accompanied by impurities when synthesized, the purification of materials forms an important part of laboratory work in chemistry. Four general separation procedures are used frequently in organic work in the laboratory and in industry: distillation, chromatography, crystallization, and extraction. The process used in any particular case depends upon the characteristics of the substance to be purified and the impurities to be removed. In order to select the most appropriate process and to employ it effectively it is important that the student understand the principles involved as well as the correct methods of manipulation. Simple distillation is one of the most commonly used purification methods.

If one compound is much more volatile than the other, the compounds can be separated in one vaporization step. Such a step is called simple distillation and uses an apparatus that consists of only a pot, a distilling head, a condenser, and adapter, and a receiver (see Figure 1). When the boiling points of two compounds differ by less than 40ºC, they cannot be separated by simple distillation. Fractional distillation, a process that has the effect of many simple distillations, must be used. A fractional distillation apparatus includes a fractionating column placed between the pot and the distilling head (see Figure 2). Typically, any one of a variety of materials, including glass beads and metal sponge, fill the fractionating column.

Figure 1. Simple Distillation Apparatus.

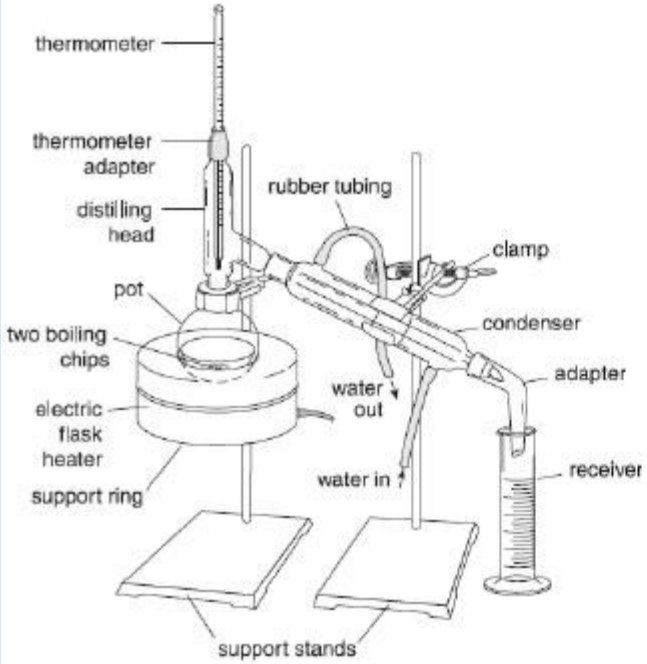
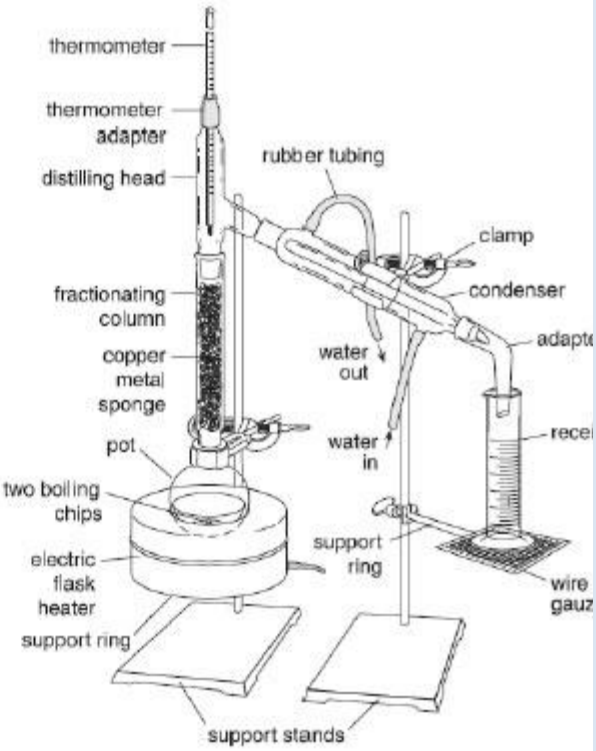


Figure 2. Fractional Distillation Apparatus



The **boiling point** of a liquid is defined as the temperature at which the equilibrium vapor pressure of the liquid equals the existing atmospheric pressure. The atmospheric pressure measured in mmHg is the current atmospheric pressure in the laboratory.

The **normal boiling point** is the temperature at which the vapor pressure of a liquid equal 760 mmHg. This is the atmospheric pressure at sea level. This is a reference value. Generally boiling points go down as the atmospheric pressure decreases, or the pressure above them in their container.

The boiling point of a liquid is a physical property of that substance and is used to help identify it, or to determine its purity. Measurement of boiling point can be done in a number of ways. One tactic that may be used to purify a liquid is to **distill** it. The process is common and one that is often used in the chemical laboratory. This exercise will distill a single liquid, and a mixture of liquids. The procedure that will be employed is called **simple distillation**.

Boiling points for a large number of liquids can be found in the **Handbook of Chemistry and Physics**. This reference book is available in the laboratory and the library. Boiling points are often listed with a superscript that indicates pressure. If no reference is given, it is assumed to be at 760 mm Hg.

Simple Distillation: A simple distillation apparatus is shown in Figure 1. This consists of a round-bottomed flask connected by means of a distillation adapter to a water-cooled condenser. A thermometer is held in place in the vertical arm of the distillation adapter by a special rubber

connector at a height adjusted so that the top of the thermometer bulb is 5 – 10 mm below the opening of the side-arm. A (vacuum) adapter is connected to the lower end of the condenser.

The distilled liquid is collected in a clean, dry receiver, commonly an Erlenmeyer flaks or small-mouthed bottle. To reduce vapor losses and minimize fire hazards, it is desirable to insert the lower end of the adapter well into the mouth of the receiver. A distilling assembly must have an opening to the atmosphere to avoid developing a dangerously high pressure within the system when heat is applied.

Key Point: When conducting a distillation, the vapor should be richer in the lower boiling component than what you started with.

Fractional Distillation: The vapors generated in the pot rise up the fractionating column and encounter cooler surfaces, upon which they condense. The condensed liquid is then reheated by rising hot vapors and revaproizes. This process of condensation and revaporization, shown graphically in Figure 5a and 5b, may occur again and again as the vapors rise up the column.

APPLICATIONS OF DISTILLATION

The application of distillation can roughly be divided in four groups:-× Laboratory scale

♣ Industrial scale

♣ Herbal distillate

♣ Food processing

♣ The latter two are distinctively different from the former two in that in the⎫ processing of beverages. The distillation is not used as a true purification method but more to⎫ transfer all volatiles from the source materials to the distillate.

Uses of Distillation

Distillation is used for many commercial processes, such as the production of gasoline, distilled water, xylene, alcohol, paraffin, kerosene, and many other liquids. Gas may be liquefied and separate. For example: nitrogen, oxygen, and argon are distilled from air.

Types of Distillation

Types of distillation include simple distillation, fractional distillation (different volatile 'fractions' are collected as they are produced), and destructive distillation (usually, a material is heated so that it decomposes into compounds for collection).

Simple Distillation

Simple distillation may be used when the boiling points of two liquids are significantly different from each other or to separate liquids from solids or nonvolatile components. In simple distillation, a mixture is heated to change the most volatile component from a liquid into vapor.

The vapor rises and passes into a condenser. Usually, the condenser is cooled (e.g., by running cold water around it) to promote condensation of the vapor, which is collected.

Steam Distillation

Steam distillation is used to separate heat-sensitive components. Steam is added to the mixture, causing some of it to vaporize. This vapor is cooled and condensed into two liquid fractions. Sometimes the fractions are collected separately, or they may have different density values, so they separate on their own. An example is steam distillation of flowers to yield essential oil and a water-based distillate.

Fractional Distillation

Fractional distillation is used when the boiling points of the components of a mixture are close to each other, as determined using Raoult's law. A fractionating column is used to separate the components used a series of distillations called rectification. In fractional distillation, a mixture is heated so vapor rises and enters the fractionating column. As the vapor cools, it condenses on the packing material of the column. The heat of rising vapor causes this liquid to vaporize again, moving it along the column and eventually yielding a higher purity sample of the more volatile component of the mixture.

Vacuum Distillation

Vacuum distillation is used to separate components that have high boiling points. Lowering the pressure of the apparatus also lowers boiling points. Otherwise, the process is similar to other forms of distillation. Vacuum distillation is particularly useful when the normal boiling point exceeds the decomposition temperature of a compound.

Principles of Steam Distillation

Steam distillation removes contaminants from water to make it essentially inert. Laboratories and technicians use distilled water for this reason, as it doesn't add anything to the component being tested. Distilled water does not have any minerals in it, which makes it unsuitable for drinking, but good for aquariums, essential oil extraction, scientific experiments and more.

TL;DR (Too Long; Didn't Read)

The process of steam distillation separates the substances of a mixture through evaporation, which then involves condensing the vapor back into liquid, taking advantage of the fact that different elements or compounds have different boiling points. It has wide uses, from water purification to extracting oils from organic matter and refining crude oil.

Reasons for Steam Distillation

Traditional distillation techniques require direct heating of the mixture to evaporate its contents. While this works well for most inorganic solutions and a few organic ones, there are many organic compounds that decompose at high temperatures, including many natural essential oils and aromatic compounds. To ensure the needed organic compounds do not get destroyed during steam distillation, technicians distill these compounds at lower temperatures.

Vapor Pressure

Matter surface has high energy molecules in contact with the atmosphere, which exert a certain pressure against the atmosphere due to their internal energies, known as vapor pressure. If this pressure exceeds the atmospheric pressure, those molecules evaporate. Since heating increases the internal energy of those molecules, it also increases vapor pressure.

How It Works

Most complex organic compounds don't dissolve in water but form a mixture instead, which separates if allowed to settle as the water settles down and the organic compounds float on top. The steam distillation process works on the principle that when a mixture of two or more undissolved liquids are heated, while ensuring that the surfaces of both liquids are in contact with the atmosphere, the vapor pressure exerted

by the system increases. This is because it now becomes the sum of the vapor pressures of all of the components of the mixture combined together. This allows for evaporation of elements with high boiling points at much lower temperatures merely by allowing them to form a mixture with water.

Extraction Procedure

Steam passes through the organic matter that contains the compounds for separation. The steam condenses against that matter to form a mixture. That mixture gets heated further by more incoming steam, which continues to pass through the matter, evaporating the mixture. Due to the reduced vapor pressure, the required organic compounds also evaporate as a part of the mixture and are thus extracted from the organic matter.

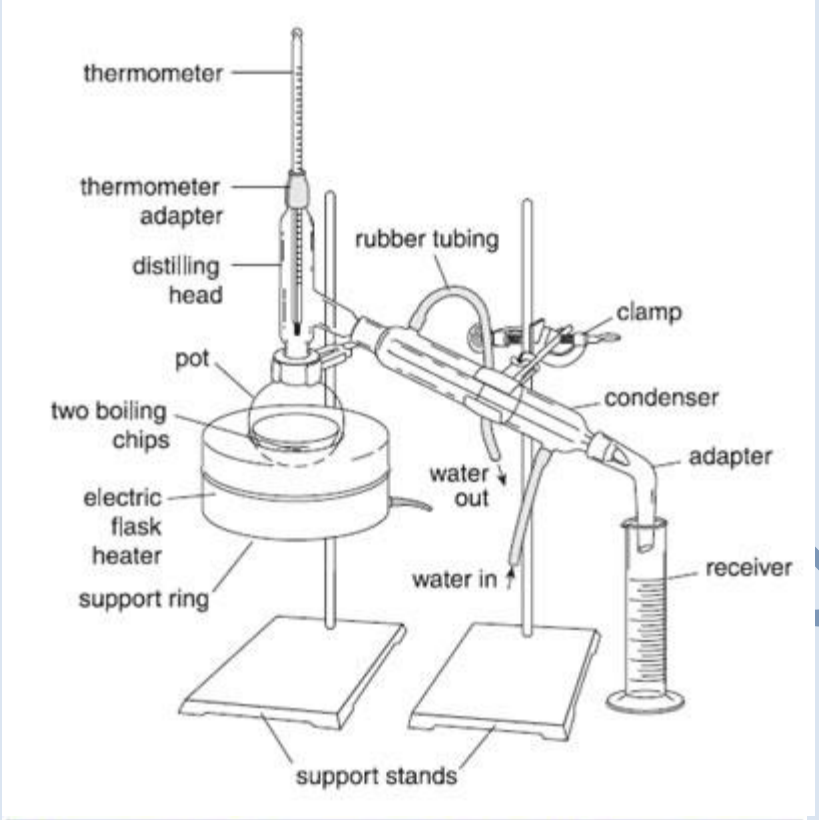
Separation Procedure

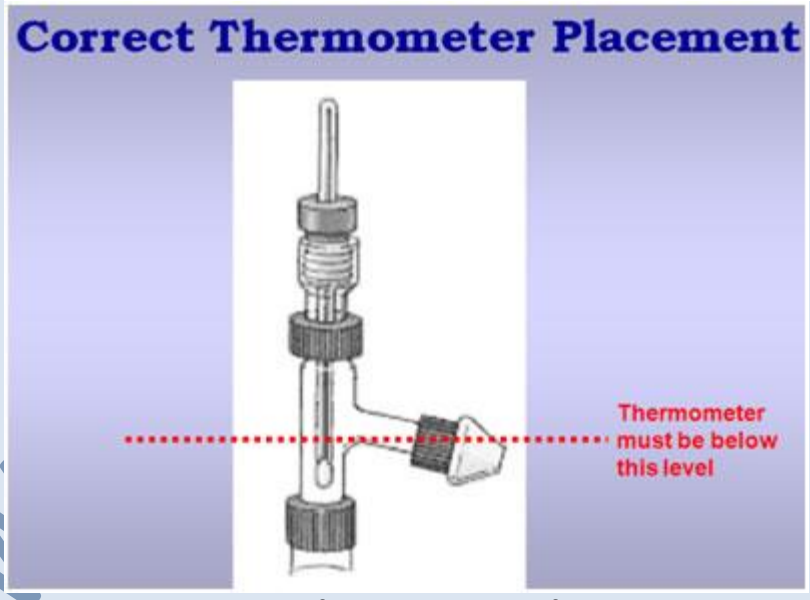
The evaporated mixture of steam and the organic compounds passes through jackets that have cold water coming in at one end. The evaporated mixture then passes out as hot water from the other end after cooling the mixture down. This condenses the mixture, which is then collected and allowed to settle. During the settling process, the extracted organic compounds come to the top, and they are then separated by filtering out the settled water from below.

PROCEDURE

REPORT AND DATA COLLECTION - The collection and graphing of data will be done on a spreadsheet. Download the Excel file to collect your data. No formal lab report is required for this experiment.

EXERCISE 1.1 Distillation of a Pure Compound by Simple Distillation
Assemble the simple distillation apparatus. Run cooling water through the condenser in at the "bottom" and out at the "top". Support the round bottom with a securely applied clamp, use a small heating mantle for heat and collect the distillate in a 25 ml graduated cylinder. Clamp all of the glassware to the back of the laboratory hood.





In the dry round bottom flask add 25 mL of methanol (containing a red dye). Add one or two tiny boiling chips, attach the boiling flask, and make certain that all connections are tight. Arrange a graduated cylinder to serve as the receiver. Heat the flask gently to boiling, and record the temperature when the first drops of distillate collect in the condenser. As the liquid boils, watch for the condensation line of vapor as it moves up the distilling head. To observe and record an accurate temperature reading, the entire thermometer bulb must be immersed in vapor. Adjust the heater to produce distillate at

a rate that is no greater than one drop per second.

Continue to distill the liquid slowly (not over 2 mL per minute) and record the distilling temperature at regular intervals during the distillation. Discontinue the distillation (and turn off the heat) when all but 2-3 mL of the liquid has distilled. Record the temperature range from the beginning to the end of the distillation; this is the observed boiling point. Note in the spreadsheet if the boiling point differs from the literature value. Allow the pot to cool for a few minutes, then turn off the water to the condenser.

Advantages of Distillation

It is an efficient method of water softening for smaller purposes.

It is relatively cheap.

It can also be reused.

Disadvantages of Distillation

Some of the unwanted elements may be found in the distilled water. Unfortunately, liquids such as herbicides which have a boiling point of 100% which is equal to that of water will tend to condense with water and therefore, separation of this two can be tough.

As a process of water softening, distillation requires a keen eye, so that unwanted elements do not mix with water.

When distillation is done on a larger scale, a very high amount of energy needed.

The distilled water does not contain any oxygen and is also very tasteless.

It has very high levels of acidity.

Conclusion

Distillation should not be considered as a method of water softening because one might end up having unwanted elements in the distilled water. For example, liquids that have a boiling point equal to water will not be separated but will condense with the water. Moreover; the process requires a lot of energy to be carried out.

REFERENCES

1. www.google.com
2. www.wikipedia.org
3. www.studymafia.org