Experiment 5

The Essential Oils of Plants and Steam distillation

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Steam distillation is a technique used for separation of compounds (solids and liquids) from complex mixtures by taking advantage of their volatility in steam.

A compound must satisfy three conditions to be successfully separated by steam distillation. It must be stable and relatively insoluble in boiling water, and it must have a vapor pressure in boiling water of the order of 10-15 mm Hg.

If **two or more compounds** satisfy these three conditions, they will generally not separate from each other but will be separated from everything else.

Normal distillation of essential oils, for example, would need quite high temperatures. On the whole these tend to be big molecules. Quite a lot of molecules of this sort will decompose by heating at high temperatures. Distilling them in the presence of water avoids this by keeping the temperature low.

Steam Distillation-The Principle

The steam distillation process works on the **principle** that when a **mixture of two or more immiscible liquids** is heated while ensuring that the surfaces of both liquids are in contact with the atmosphere, the **vapor pressure** exerted by the system is increased.
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.

When two miscible liquids A and B (ideal solution), are distilled, the ideal solution follow **Raoult's Law**:

$$P_{total} = P°_A \times N_A + P°_B \times N_B \ , \ (\text{observed } P_A = P°_A \ N_A)$$

Where $P°_A$ = vapor pressure of pure A, $P°_B$ = vapor pressure of pure B
N_A = mole fraction of A and N_B = mole fraction of B

Thus, the **composition of the vapor** will depend on both the **vapor pressures** and the **mole fractions** of each component.

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In contrast, when two **insoluble** (immiscible) liquids are "mixed" to give a **heterogeneous** mixture, each exerts its own vapor pressure, independently of the other:

$$P_{total} = P°_A + P°_B$$

The mole fraction term does not appear in this equation, because the compounds are not miscible. The total pressure above the mixture is simply the **sum of the vapor pressures** of the pure liquids $P°_A$ and $P°_B$ at a given temperature..
When the total pressure equals 760 mm Hg, the mixture boils.
The composition of the vapor from an immiscible mixture, in contrast to the miscible mixture, is determined only by the vapor pressures of the two substances codistilling. Thus, the composition of the vapor from an immiscible liquids can be expressed as :

$$\frac{\text{Moles A}}{\text{Moles B}} = \frac{P°_A}{P°_B}$$

In steam distillation, the two components (water and organic) behave as distinct entities.

$$P_{total} = P°_{water} + P°_{organic}$$

If one is to take the ratio of a gas law written for the gaseous water and one written for the organic gas, the following expression is obtained:

$$\frac{P_{H_2O} V_{H_2O}}{P_{Org} V_{Org}} = \frac{N_{H_2O} RT_{H_2O}}{N_{Org} RT_{Org}}$$ this equation reduces to $$\frac{P_{H_2O}}{P_{Org}} = \frac{N_{H_2O}}{N_{Org}}$$

R, T, V: constants

N_{Org} = Moles$_{Org}$ = Weight$_{Org}$ / Molecular Weight$_{Org}$

$$\frac{Wt_{Org}}{Wt._{H_2O}} = \frac{\text{Vapor pressure}_{Org} \times \text{mol. Wt.}_{Org}}{\text{Vapor pressure}_{H_2O} \times \text{Mol. Wt.}_{H_2O}}$$

Some other applications of steam distillation

Steam distillation can be used to extract some **natural products** - for example, to extract

eucalyptus oil
from eucalyptus

citrus oils
from lemon or orange peel

perfumes
Ex: Jasmine

Experimental Procedure:Part I:

1) Set up a steam distillation **apparatus** as shown below. Place your separatory funnel filled with distilled water in the two-neck adapter.

2) Obtain about **15g spice**. If necessary, grind the spice in a mortar and pestle. Add the ground spice to your **500 mL round-bottom flask.**

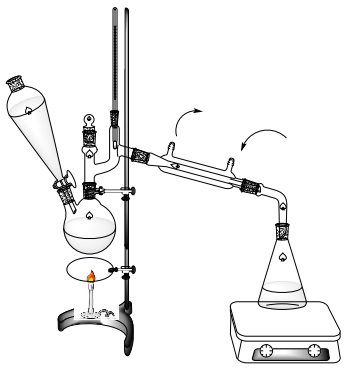
3) Fill the flask **half full** with distilled water. Add a couple of boiling chips. The round-bottom flask with the spice mixture will be the distilling flask.

4) Heat the distilling flask slowly using a heating mantle or Bunsen burner. You may **add water** sparingly from your separatory funnel so the slurry inside **doesn't dry** out and burn.

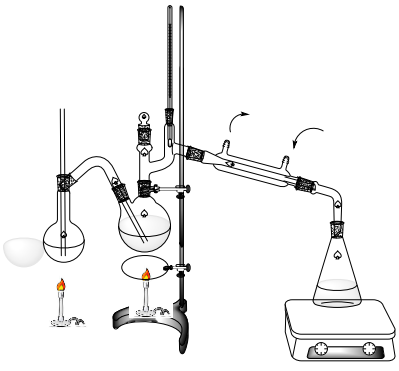
5) Stop the distillation when you have about **100 mL of distillate**, or you have been collecting distillate for one hour.

6) Record the volume of your distillate.

Steam distillation-Modified Setup



Direct steam distillation



Steam distillation using live steam

Part II Extraction of the Essential Oil:

•Extract the distillate twice with 15 mL aliquots of dichloromethane.
a. add distillate to separatory funnel,
b. add 15 mL of CH_2Cl_2,
c. shake,
d. drain lower layer,
e. repeat **steps b**. through **d**.
Combine and save the organic (dichloromethane) layers.

•Next, extract the combined dichloromethane layers twice with 15 mL aliquots of a saturated NaCl solution.

a. add organic solution to separatory funnel,
b. add 15 mL of NaCl solution,
c. shake,
d. drain lower layer and save,
e. drain upper layer and discard,
f. repeat **steps a**. through **e**.

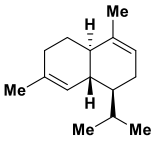
Part II Extraction of the Essential Oil:

•Use drying agent such as $CaCl_2$to dry the organic layer. Filter off the drying agent. Collect the filtrate in a pre-weighed beaker.

•Remove the solvent by heating over a **hot water bath** in the fume hood. Use a boiling stick to facilitate boiling. Do not heat to dryness – let the final bit of solvent evaporate with beaker off the hot water bath.

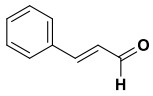
•Obtain the mass of your product.

Analysis: Record the IR spectrum of the essential oil or GCMS.



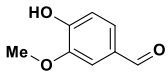
δ-Cadenine

Trigonella
الحلبة



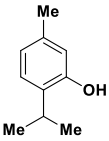
cinnamaldehyde

Cinnamonقر
فة



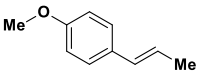
vanillin

Vanilla beans
فانيلا



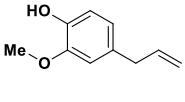
Thymol

Thyme
زعتر



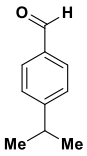
anethol

Anise
يانسون



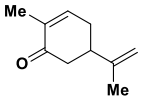
eugenol

Cloves
كبش قرنفل



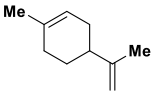
Cuminaldehyde

Cumin
كمون



carvone

Carawy
كراوية



limonene

Orange
peels
قشر البرتقال