EXPERIMENT 7

The essential oils of plants
Steam distillation

The Essential Oils of Plants and Steam distillation

- Steam distillation is a technique used for separation of compounds (solids and liquids) from complex mixtures by taking advantage of their volatility in steam.
- When steam is used to provide one of the immiscible phases, the process is called **steam distillation**.
- The advantage of this technique is that the desired material distills at a temperature below 100°C.
- A compound must satisfy three conditions to be successfully separated by steam distillation:

1. it must be stable
2. relatively insoluble in boiling water
3. must have a vapor pressure in boiling water of the order of 10-15 mm Hg.

The Essential Oils of Plants and Steam distillation

- The advantage of this technique is that the desired material distills at a temperature below 100°C. Thus, if unstable or very high-boiling substances are to be removed from mixtures, decomposition is avoided.
- Normal distillation of essential oils would need quite high temperatures. 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.
- 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 its pressure 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.

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Differences between Distillation of Miscible and Immiscible Mixture

- 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.

- 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.

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- The composition of the vapor from an immiscible mixture, in contrast to that of the miscible mixture, is determined only by the vapor pressures of the two substances codistilling. The below equation defines the composition of the vapor from an immiscible mixture.

$$\frac{\text{Moles A}}{\text{Moles B}} = \frac{P^0_{\text{A}}}{P^0_{\text{B}}}$$

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

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

$$\frac{\text{Moles substance}}{\text{Moles water}} = \frac{P^0_{\text{substance}}}{P^0_{\text{water}}}$$

$$\frac{\text{Wt substance}}{\text{Wt water}} = \frac{(P^0_{\text{substance}})(\text{Molecular weight}_{\text{substance}})}{(P^0_{\text{water}})(\text{Molecular weight}_{\text{water}})}$$

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- When the total pressure equals 760 mm Hg, the mixture boils.

Sample Calculations for a Steam Distillation :

Problem How many grams of water must be distilled to steam distill 1.55 g of 1-octanol from an aqueous solution? What will be the composition (wt%) of the distillate? The mixture distills at 99.4°C.

Answer The vapor pressure of water at 99.4°C must be obtained from the CRC Handbook (= 744 mmHg).

a. Obtain the partial pressure of 1-octanol.

$$P^{o}_{\text{1-octanol}} = P_{\text{total}} - P^{o}_{\text{water}}$$

$$P^{o}_{\text{1-octanol}} = (760 - 744) = 16 \text{ mmHg}$$

b. Obtain the composition of the distillate.

$$\frac{\text{wt 1-octanol}}{\text{wt water}} = \frac{(16)(130)}{(744)(18)} = 0.155 \text{ g/g-water}$$

c. Clearly, 10 g of water must be distilled.

$$(0.155 \text{ g/g-water})(10 \text{ g-water}) = 1.55 \text{ g 1-octanol}$$

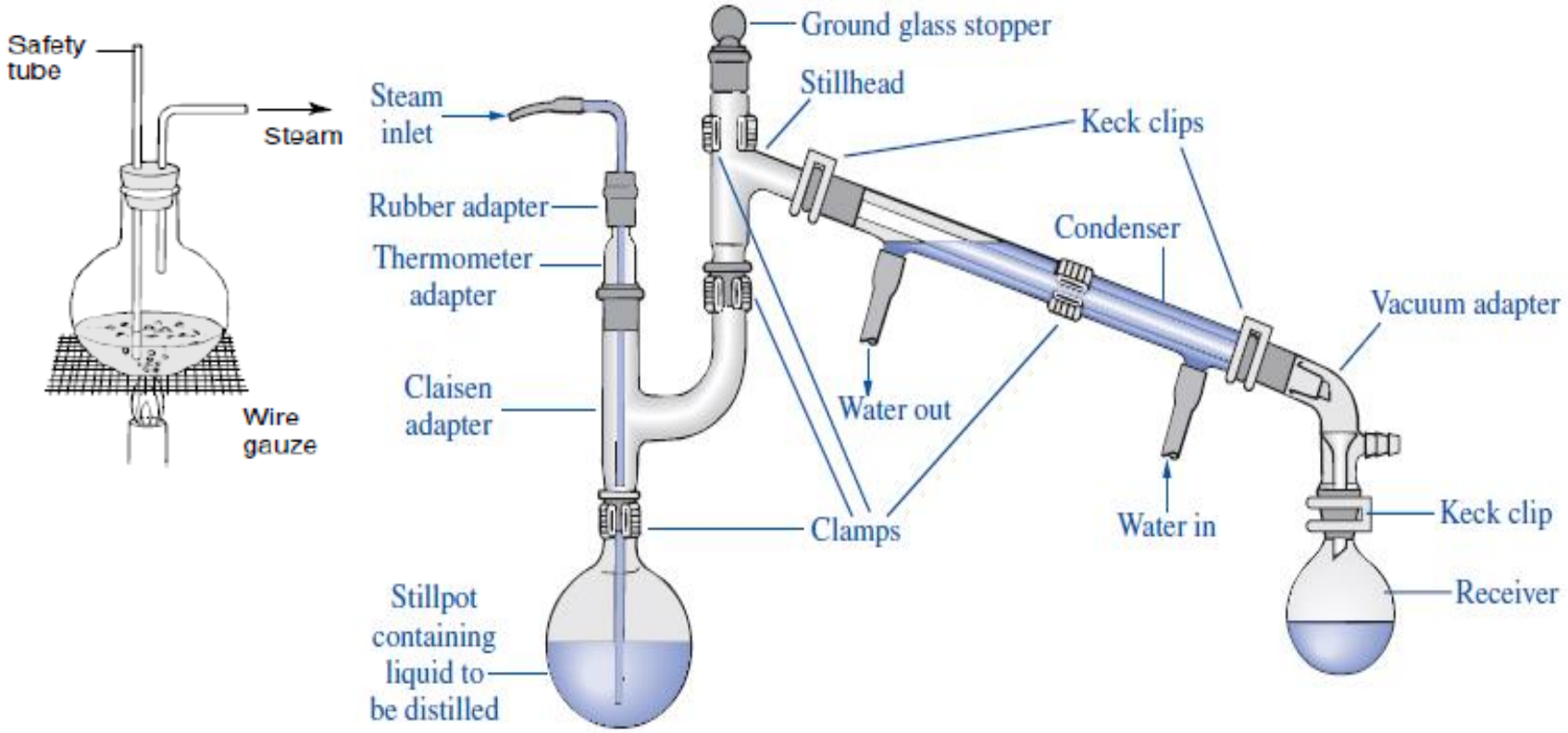
d. Calculate the weight percentages.

$$\text{1-octanol} = 1.55 \text{ g}/(10 \text{ g} + 1.55 \text{ g}) = 13.4\cdots$$

$$\text{water} = 10 \text{ g}/(10 \text{ g} + 1.55 \text{ g}) = 86.6\cdots$$

The Essential Oils of Plants and Steam distillation

Types of steam distillation: 1. Steam distillation using live steam



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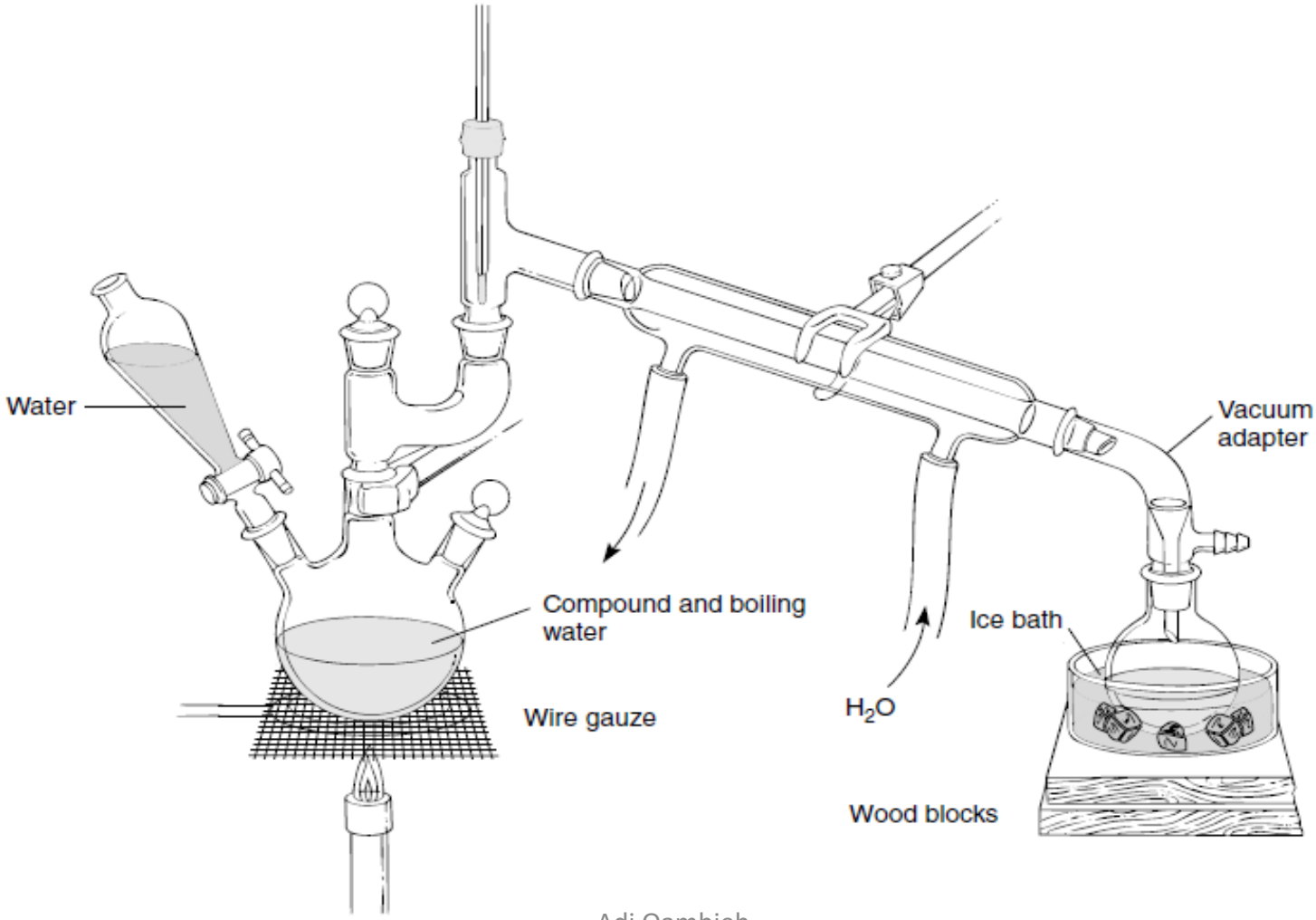
Types of steam distillation: 2. Direct steam distillation



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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…..etc, and to extract oils used in perfumes from various plant materials.

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Experimental Procedure:

1. Place 20 gram of the selected ground spice, in a 1000 ml (3-neck) round bottom flask
2. Fill the flask half full with distilled water.
3. Add a couple of boiling chips. The round-bottom flask with the spice mixture will be the distilling flask.
4. Assemble the apparatus for a direct steam distillation.
5. Heat the distilling flask slowly using a Bunsen burner.
6. Stop the distillation when you have about 150 mL of distillate, or you have been collecting distillate for one hour.
7. Pour the distillate into a 250 ml separatory funnel.
8. Extract it twice, using 15 ml portions of methylene chloride. After shaking each time, separate the lower methylene chloride into a 50 ml Erlenmeyer flask.
9. Dry the combined extracts over a small amount of anhydrous sodium sulfate(about 1 gram).

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10. Filter off the drying agent.
11. Collect the filtrate in a pre-weighed 100 ml beaker.

12. Remove the solvent by heating over a hot water bath in the fume hood.
13. Do not heat to dryness – let the final bit of solvent evaporate with beaker off the hot water bath.

14. Record the weight , calculate percentage yield and write the structure of the main essential oil in the selected spice.

- **List of spices provided for this experiment include:** *Thyme, Caraway seeds, Cumin seeds, Ground gloves, cinnamon sticks , Anise.*

- **Yield of the oil obtained is usually low**, based on the amount and the type spice used

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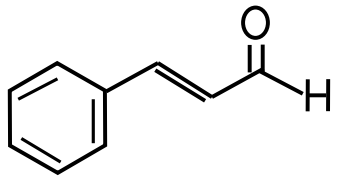

Cinnamaldehyde

(Cinnamon)

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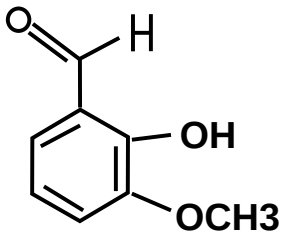

Vanillin

(Vanilla Beans)

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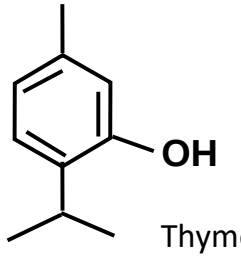

Thymol

(Thyme)

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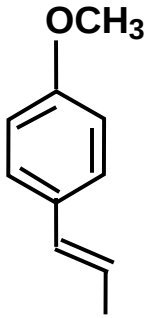

Anethol

(Anise)

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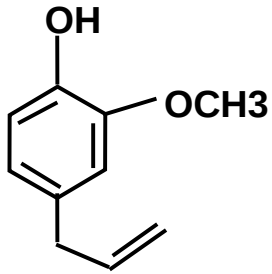
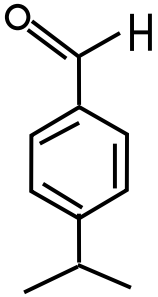


Eugenol

(Cloves)

كبش قرنفل



Cuminaldehyde

(Cumin)

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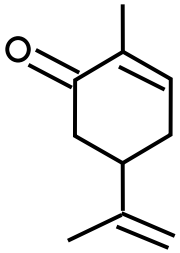

Carvone

(Caraway)

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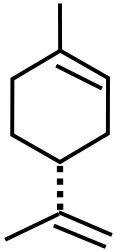

Limonene

(Orange Peels)

قشور البرتقال