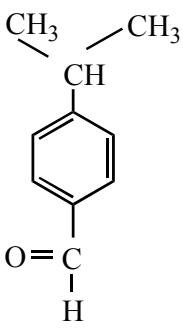
ISOLATION OF ESSENTIAL OILS
BY STEAM DISTILLATION

PURPOSE

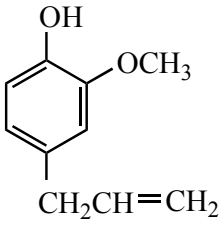
Recover essential oils from common spices using steam distillation.

THEORY- ESSENTIAL OILS

The aroma of many naturally occurring plants is due to volatile oils present at low levels in this vegetation. These essential oils make up the characteristic odor of many plants, among them, eucalyptus, citronella, garlic, oranges, roses, peppermint, and many more. Unfortunately, many of these oils decompose when heated at higher temperatures. Essential oils can be removed readily from natural substances by use of steam distillation. The following spices will be handled by you or some of your classmates to remove their essential oil: fennel, cumin, cloves, cinnamon, caraway, and allspice. Each of these spices will provide one of the compounds shown below:



A



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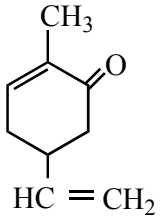

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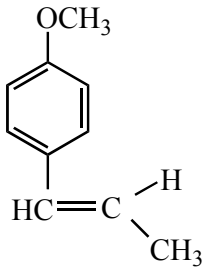
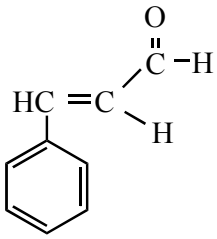


D



E

THEORY- STEAM DISTILLATION

Boiling occurs when the vapor pressure of a liquid equals the atmospheric pressure. When we mix water with a material that is INsoluble in the water, the two substances act independently of each other. Each substance contributes vapor pressure toward equaling the atmospheric pressure. These added vapor pressures make it easier to equal atmospheric pressure so less heating is required. Steam distillation can allow distillation of organic materials at lower than normal temperatures. This is especially useful when substances are prone to decomposing under higher temperatures.

PROCEDURE

Note: You need not prepare physical property data in your notebook for the spices.

1) Your instructor will issue you a spice. Record the identity of the spice you receive and describe its odor. Weigh out approximately 10 grams of the spice. Grind the spice using a

mortar and pestle, then pour the ground spice into a prewighed beaker so as to obtain the exact mass.

2) Pour the ground spice into a 250 mL roundbottom, add about 80 mL of water and a boiling stone. The spice should soak for at least 15 minutes as you set up your distillation apparatus.

3) Set up your spice-filled roundbottom for simple distillation. Since you will be using a 100 mL roundbottom as a receiving flask, you will need to use a curved vacuum adapter in between this receiver and the end of the condenser (see Fig 20. 1, p. 168 Survival Manual) Clamp the vacuum adapter!

4) Initiate heating with your heating mantle, send cooling water through your condenser, and capture the cloudy distillate. You might have to cut back on the heat if the spice starts to froth and threaten to push into the condenser. Try to collect at least 20 mL of distillate. WHEN CLEANING UP, DO NOT FLUSH SPICES DOWN THE DRAIN! The instructor will provide a bucket for flask cleanout.

5) Shut down the steam distillation and transfer the distillate into a small separatory funnel. Extract with 10 mL of methylene chloride. IF THE TWO LAYERS FAIL TO SEPARATE, PERFORM THE FOLLOWING: Add 2 mL of saturated NaCl solution and swirl gently. Be aware that saturated sodium chloride solutions can be quite dense and just might switch places with the methylene chloride layer normally on the bottom. Test the layers if you are not sure using techniques we have already used in previous labs.

6) After the layers separate, transfer the organic layer into a clean, dry Erlenmeyer flask. Repeat this extraction on the aqueous layer with another 10 mL of methylene chloride, adding the organic layer to the Erlenmeyer. If there are any visible drops of water in your flask at this point, transfer the organic layer to another clean flask while leaving the water drops behind.

7) Dry the organic layer with about 2 grams sodium sulfate for a period of 10-15 minutes.

8) Weigh a larger size test tube. Decant a portion of the dried organic layer into the tube and use a stream of air to evaporate the solvent. Add the remaining organic layer when room permits so its solvent can be evaporated. Do not allow the drying agent to enter the test tube. (We will <u>not</u> use a rotoevaporator to remove solvent for fear of degrading the essential oil through excessive heat.)

9) Reweigh the test tube and determine the mass of recovered oil. Calculate the % recovery of oil from the original amount of spice used.

ANALYSIS

10) Use the infrared spectrophotometer to obtain an "IR" of your oil. Be sure to have the software mark the important absorption peaks. Print your IR. YOU NEED NOT SUBMIT A SAMPLE. They can be rinsed down the drain after the instructor approves your IR spectrum.

CONCLUSION

Examine the structures of the possible essential oils you may have recovered (see structures A-E). Use structural characteristics and IR absorptions to determine which oil you isolated. You should develop a matrix like the one shown below. Use the structures shown to complete the table. You need only use "YES" or "NO" to complete each grid.

Your conclusion should clearly explain what absorption was due to which feature of your oil. You should discuss each structure (A-E) and explain why each could be or could NOT be your oil. Report all other pertinent information in your Conclusion as well.

	Aldehyde CHO	Phenolic OH	Ketone C=O	Phenyl Ring	Alkene
Cmpd A					
Cmpd B					
Cmpd C					
Cmpd D					
Cmpd E					

QUESTIONS

1) When the freshly distilled material reached the receiver flask, it looks cloudy. Why?

2) Why does distilling an essential oil using steam help avoid decomposition?

The Professor says, "This is ESSENTIALLY our last lab!"