Organic Chemistry Laboratory
#8 - Steam Distillation of Essential Oils

Plants have characteristic aromas because of volatile oils contained in every part of the plant. These oils are often concentrated in certain parts of the plant such as the seeds, bark, or flowers. These oils give a plant its particular essence thus the term essential oil. The essential oils of various plants have been used for centuries as flavorings, fragrances, and medicines. Some essential oils, such as limonene, are common to many different plants while others, such as eugenol, are more specific to a particular species of plant. In the case of clove and cinnamon the flavor is primarily due to one compound (eugenol and cinnamonaldehyde respectively) although the full flavor is due to a mixture of several compounds.

The isolation of essential oils is sometimes accomplished by steam distillation. In this technique, compounds that are less volatile than water are co-distilled with water. This allows for distillation at temperatures lower than necessary to distill the oil by itself. High temperatures will often cause an organic compound to decompose. In this experiment you will steam distill the essential oils from cloves.

Pre-lab Reading:

Read pages 183-187 in Chapter 20 (pay attention to figure 20.14 as this is very similar to what you will set up in lab), pages 282-289 in Chapter 34 and pages 321-322 of the Zubrick text.

Pre-lab Assignment:

You will purify eugenol (see fig. 1) from the essential oil of cloves by dissolving the oil in ether and then wash with 10% NaOH. This takes advantage of the fact that phenols are slightly acidic and will react with strong bases to produce a water-soluble salt. Once removed from the ether, the salt can be acidified and extracted from the aqueous layer with fresh ether. Write the correct acid/base reaction for this conversion of eugenol to its sodium salt. Also, circle and identify the main functional groups of eugenol.

Procedure

Obtain 10 g of a freshly ground spice and record the exact mass of the spice. Carefully transfer the ground spice into a 500 mL round bottom flask and add 150 mL water and a boiling stone. With a grease pencil, mark the flask at the level of the water inside. Assemble the steam distillation apparatus following the example provided in the lab. Add water to the addition funnel above the distillation flask. *Have your assembled apparatus checked by the instructor or teaching assistant before applying heat!* Since your distillate is primarily water it is safe and convenient to use a Bunsen burner as a heat source. Using a 125 Erlenmeyer flask as the receiver, distill 100 mL of distillate. Occasionally, you will have to add water to the distillation flask by opening the stopcock on the addition funnel; use the mark you made earlier as a guide.

Pour the distillate into a separatory funnel and add 10 mL of methylene chloride to the receiver and add this to the funnel. Shake the funnel to mix thoroughly and place in a ring stand to allow the layers to completely separate. The extraction should be carried out in the hood to avoid contact with the vapors of methylene chloride. Drain the organic layer into a 25 or 50 mL Erlenmeyer flask. Extract the aqueous layer once more using 5 mL of fresh methylene chloride. Drain the organic layer into the flask carefully. Analyze the methylene chloride solution using a TLC plate and the solvents provided (10% ethyl acetate/hexanes). You should set aside a small sample (~0.5 mL) of this organic layer for later analysis. Transfer the remaining aqueous layer into a beaker or flask and set aside until you are certain that you are done with it.

Place the methylene chloride solution in the separatory funnel and extract it with three portions of 5% sodium hydroxide solution. Transfer the methylene chloride to an Erlenmeyer flask and dry over anhydrous sodium sulfate. Decant the methylene chloride into a clean, dry, tared flask and evaporate the solvent. When the liquid in your flask stops boiling (i.e., no more bubbling at all), remove it from the hot plate and allow it to cool. When cool, and dry on the outside, weigh the flask and determine the weight of the oil collected and calculate the percent yield based on the mass of the spice used.

The combined sodium hydroxide extracts are made acid to litmus by adding hydrochloric acid. The acidic solution is then extracted with methylene chloride (3×25 mL). The combined organic extracts are dried with sodium sulfate, filtered, and evaporated to dryness as before. Record the mass of the residue and percent yield based on spice used.

Examine solutions of the two residues and the original extract by TLC. Obtain an IR spectrum for each of the residues. You can find a copy of an IR of eugenol in the library to compare against your sample. Comment on the success of your separation, i.e. how pure is your eugenol sample?

Report:

Report the weight of the spice, the weight of the oil obtained and the percentage of oil in spice. Include any observations and conclusions based on the TLC and IR spectra of your samples.

Questions:

1. A mixture of water and nitrobenzene distills at 99.3 °C when the barometric pressure is 760 mm Hg. The vapor pressure of water at 99.3 °C is 740 mm Hg. Calculate the weight ratio of water to nitrobenzene that will distill.

2. When an unknown liquid is steam distilled at 93 °C (vapor pressure of water at this temperature is 589 mm) and 760 mm, the distillate contains 6.0 g of water and 11.0 g of oil. Calculate the molecular weight of the oil.

Steam Distillation

Data Report Sheet

Name: ___________________________

Section: __________________________

Date: ____________________________

Mass of spice used	_____________________g
Mass of eugenol recovered	_____________________g
Mass of eugenol acetate recovered	_____________________g
Percent yield of eugenol recovered	_____________________%
Percent yield of eugenol acetate recovered	_____________________%
R_f of eugenol	_____________________
R_f of eugenol acetate	_____________________

Prelab: Steam Distillation

Name:_____________________

Lab section:___________

Date:__________

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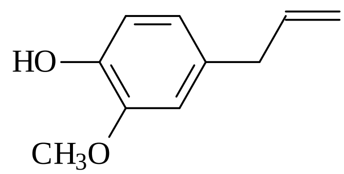

Fig. 1: Eugenol