

## Steam Distillation

This experiment involves an extraction of a natural product using the technique of steam distillation. The principle component of oil of cloves is an aromatic compound, eugenol, which you will isolate and identify by infrared spectroscopy and thin layer chromatography (TLC). During the first week, you will obtain the oil of cloves and do a TLC analysis on it. During the next lab period, you will take an infrared spectrum of your oil to complete the experiment.

### New techniques:

Steam distillation  
Thin-layer chromatography  
Infrared spectroscopy

### Reading (in Pavia):

Steam distillation: chapter 18, pp. 278-9; 283-4.  
Thin layer chromatography: chapter 19, pp. 286-295; chapter 20, pp. 313-325.  
Infrared spectroscopy: chapter 25, pp. 369-373; 379; 385-407 (see also your lecture text, if necessary)

### Before lab:

- 1) Determine the structure and boiling point of eugenol.
- 2) Find the density and boiling points of dichloromethane and ether and their miscibility with various solvents. In an extraction, would you expect to find dichloromethane as the top or bottom layer?
- 3) What would be the principal IR absorption frequencies for eugenol? That is, what would you look for to identify it?

### Steam distillation/Extraction:

This experiment involves the microscale apparatus. Place about 0.4 g (record exact amount) of ground cloves into a 10 mL round bottom flask and add 4 mL of water and a boiling chip. Assemble a steam distillation apparatus with a Hickman head (with septum cap over the collection port) and a water-cooled reflux condenser as pictured on page 52 of your text. Heat the mixture to provide a steady rate of distillation, controlling the heat by raising or lowering the sand bath. You need to maintain steady heat for distillation, but avoid too much heat, as the clove mixture can bubble and froth into the Hickman head. *Do not* allow this to happen!

Replace any water lost in the distillation by adding water via a long Pasteur pipette through the condenser. Periodically collect the distillate in a 5 mL conical vial (vial #1) with a different pipette and rinse the walls of the distillation head regularly with small amounts of water. Keep the head capped unless you are removing distillate. Collect about 3 mL of distillate, leaving room in the vial for about 1 mL of methylene chloride for the extraction.

Extract the essential oil from the distillate with methylene chloride, adding 1 mL to vial #1, cap it, and shake vigorously (remember to vent!). Allow the layers to separate and transfer the methylene chloride layer to a second dry vial (vial #2, screw-capped or conical). Repeat the extraction with two more 1 mL portions of methylene chloride, taking care not to remove any water in your transfer of the methylene chloride. Dry your collected methylene chloride layers with sodium sulfate.

Clean and dry another 5 mL conical vial (vial #3) and weigh it accurately. Plug a short Pasteur pipette with a small piece of filter paper and filter your organic layer through this pipette into your weighed vial. Rinse vial #2 with a small amount of methylene chloride. Evaporate the methylene chloride in vial #3 by heating gently in a sand bath (in the hood), until only a drop of oily residue remains. Weigh your vial and calculate the amount of isolated oil of cloves.

Take an infrared spectrum of the oil. If you have only a very small amount of oil, dissolve it in a very small amount of methylene chloride and transfer this solution to the salt plates, allow the methylene chloride to evaporate, and take the spectrum.

*Clean up:* The material in the still pot can be thrown in the trash (after removing any water). After taking the IR spectrum of eugenol, clean your salt plates with methylene chloride according to procedure. You may wash any excess down the sink with water.

### **Thin layer chromatography**

Dissolve a small drop of your oil in a milliliter of ether. Apply a small spot of this solution to a TLC plate using a capillary tube (do not use an unaltered melting point capillary). Develop the plate in your solvent chamber (a beaker with filter paper and a watch glass lid) using the appropriate solvent system until the solvent front is just below the top of the plate. Mark the solvent front with a pencil (never a pen). Locate the spots with a UV lamp (NEVER look directly at the lamp itself). Calculate the R<sub>f</sub> value for the largest spot of your oil. Can't tell which is the largest spot? You probably spotted too heavily. Try again with a lighter spot this time. TLC methods are extremely sensitive, so you need very little compound to detect (fractions of a milligram are sufficient). Spot another plate with a solution containing stock eugenol, develop it and calculate the R<sub>f</sub> value. Compare to the R<sub>f</sub> value of your oil.

### **Infrared spectrum**

Take an IR spectrum of your oil and identify your major peaks.

**For the lab report:**

Report your percent recovery of eugenol from cloves. Include your infrared spectrum with all major peaks identified to confirm the presence of eugenol and what molecular vibrations they correspond to. That is, indicate why your IR spectrum confirms the presence of eugenol. Present you calculated Rf values for your eugenol and your oil. Also, provide an explanation of steam distillation. Why does it work?