12AL Experiment 7: Extraction of Clove Oil

Instructor Note: Day 1 (half of the class); Day 2 (other half); limited macro distillation equipment & organic students must perform on an individual basis.

Safety: Proper lab goggles/glasses must be worn (even over prescription glasses). Utilize care when working with organic solvents. As always, ask where organic waste containers are located in the lab.

Background: Essential oils are harvested from plants and spices to be used in a variety of manners – from perfumes to medicines. Essential oils are the "essence" of the plant and generally have the same odor as the plant or spice itself! Today you will be performing a natural extraction of the essential oil eugenol, the main component of cloves. Eugenol has a variety of uses including perfumes, acne products, antiseptics, flavorings, and dental analgesics. For additional information on therapeutic uses of common spices & plants see: http://www2.hawaii.edu/~johnb/micro/m140/syllabus/week/handouts/m140.8.3.html

Plants or spices are ground up to help unlock the essential oils from any bark or strong casings. Then the organic chemist generally performs a steam distillation, where the essential oil, most of which are slightly soluble in water, travels with the steam through the distillation apparatus to collection. Steam distillations are slow and long and result in a small amount of oil collected; that is why essential oils are so expensive to buy in the store.

It is also possible to synthetically produce the same essential oil through a chemical reaction – there is no difference between the essential oil produced in a synthesis or extracted naturally; the debate comes from the waste and chemicals used in chemical syntheses.

Today you will be performing a version of a steam distillation. We do not have the exact equipment required to do a steam distillation so you will be setting up your regular simple macro-distillation apparatus. After grinding of your cloves (unless ground cloves are provided), you will be boiling the ground cloves in water in your round-bottom flask. As the water boils and converts to steam, eugenol will slowly travel with the steam through the simple column and condenser where eugenol oil and liquid water will drip out into a collection beaker. Then you will perform separation techniques to remove the eugenol oil from the water. Make sure to work slowly and diligently as you are isolating a small amount of oil and do not want to repeat a long distillation.

Objective: To extract the main component of cloves through distillation and identify its structure through IR spectroscopy. To learn how essential oils can be removed from their natural sources through grinding and distilling.

Procedure:

1. Measure out ~1.0-1.5 grams of ground cloves. (If whole cloves are provided, use a mortar and pestle to grind up the cloves into coarse particles, not too fine, and then measure out 1.0-1.5 grams). Pour into a round-bottom flask.
2. Set-up your Macro Simple Distillation, but use a Bunsen burner as your heat source, and a 20-25 mL beaker as collection container. Make sure to review the set-up if necessary. Never let go of a glass distillation piece until it is securely clamped! And make sure all glass joints are lubed lightly with glycerol!

3. Add a few boiling chips to the round-bottom flask containing your cloves. Fill the flask with distilled water to the halfway mark – it is very important that you constantly maintain the water level at the halfway mark throughout the entire distillation! You can simply pipette water down the top of the simple columm even during heating.

4. Heat the mixture to a good boil, but NOT "out-of-control" boiling! If the mixture begins to froth up the column, you are heating too high. Immediately remove the burner to calm the mixture down, and then adjust the flame before putting it back under the flask.

5. Collect about 8 mL of the water/clove oil distillate. Remember, this could take awhile! You should not have any clove froth/ clove bark in your distillate.

6. Add 3.0 mL dichloromethane (methylene chloride) to your distillate. The clove oil is more soluble in dichloromethane than in water. Separate the two layers – which layer is which? Again, you need to make this decision as you have done in previous experiments. One layer is water, the other is the dichloromethane layer with clove oil – what information do you need about water and dichloromethane to decide? Do not throw any layers away just in case you make a mistake. Label your containers – aqueous or organic.

7. To the aqueous layer, add another 3.0 mL of dichloromethane in an attempt to get any remaining clove oil out of the water. Separate the layers, making sure to put the dichloromethane layer with the original dichloromethane layer.

8. Repeat step 7 again.

9. "Dry" the dichloromethane layer with a tiny amount of anhydrous powder. Remember, tiny means tiny! You just want to soak up any remaining water droplets that may remain in organic layer.

10. Decant the dichloromethane layer into a clean small beaker (don't pour over the anhydrous powder).

11. Evaporate the dichloromethane in a warm-water bath, leaving behind the clove oil in the bottom of your beaker.

12. Run an IR of your clove oil; Analyze the IR fully – all bonds must be labeled with wavenumbers on appropriate positions on IR.

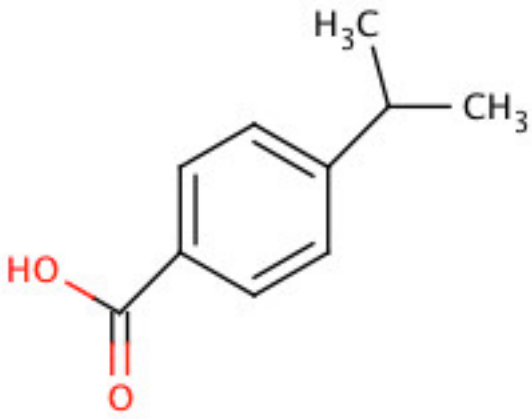
13. Determine the structure of the main component of clove oil from the 5 molecules shown in the prelab. Draw structure on your IR too. Attach to postlab.

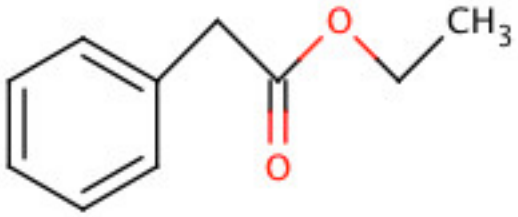
12AL Prelab Experiment 7: Isolation of Clove Oil

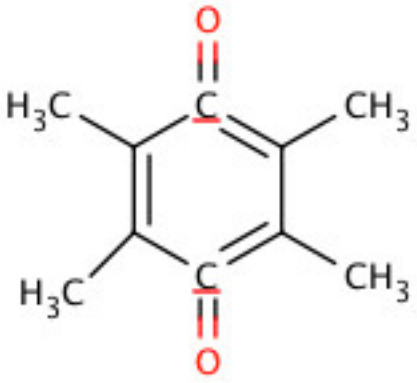
1. The following 5 molecules all have the molecular formula $C_{10}H_{12}O_2$ and you will prove which one of the following is the main component of clove oil by extraction, purification, and IR analysis.

   a. Label all the functional groups (ester, ether, aromatic, ketone, etc….) present for each molecule.

   b. List ALL unique bonds with their wavenumbers that you would see in an IR.



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C
CH3
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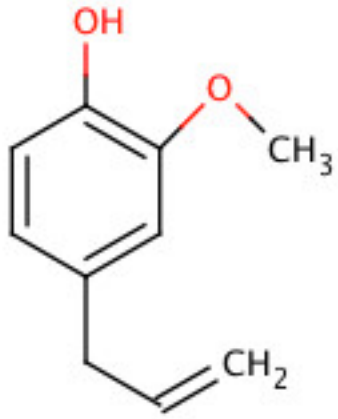
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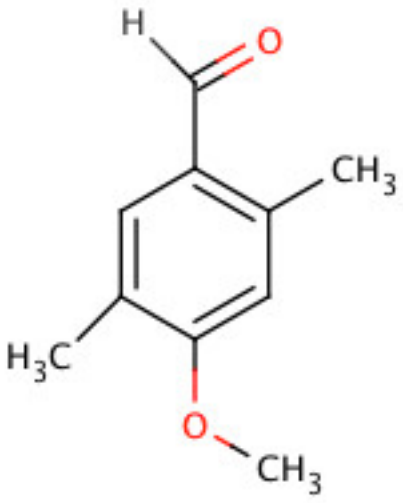
CH2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O
CH3
H3C
O
CH3

12AL Postlab Experiment 7: Isolation of Clove Oil

1. The major component of clove oil is a compound called Eugenol. One of the minor components is called Acetyleugenol. Draw the structures of both of these compounds AND then explain how you would differentiate between the two compounds using IR spectroscopy.

2. Often eugenol is extracted from organic ether using aqueous sodium hydroxide - temporarily converting it to an aqueous salt, and then aqueous hydrochloric acid is used to convert the salt back to the eugenol once all layers are separated.

a. Show the reaction *mechanism* that occurs between eugenol and sodium hydroxide.

b. Show the reaction *mechanism* that occurs between the eugenol salt and hydrochloric acid.

3. Is your completely analyzed IR spectrum attached?