Advanced Chemistry: Isolation of Cinnamaldehyde from Cinnamon Sticks Names:

Procedure:

1. Set up an immersion heater in a 400 mL beaker with 300 mL of water

2. Break up a cinnamon stick into small slivers (15 x 3 mm). Place the pieces into a tarred reaction vial and mass the cinnamon and vial. Use about 1.5-2.0 g of cinnamon. Record the mass of the vial and vial/cinnamon

3. Add 5 mL of tap water to the reaction vial and seal the flask with a Teflon-coated cap.

4. Place the vial into the hot water and check for bubbles. If bubbles appear, remove the vial and tighten the cap.

5. Heat the vial for 25 minutes. After 25 minutes, remove the vial and allow to air cool for a few minutes. After cool, place into a beaker with cold water for about three minutes.

6. Open the vial slowly. Remove the aqueous solution of cinnamaldehyde into a clean reaction vial. Do not transfer any of the cinnamon pieces. These may be discarded into the garbage.

7. Smell the mixture. Record.

STOP FOR THE DAY. CLEAN UP YOUR LAB. LABEL YOUR VIAL CLEARLY AND SET ASIDE FOR TOMORROW.

8. Extract the cinnamaldehyde with ethyl ether. Add about 2-3 mL of ethyl ether to the aqueous solution. Tightly cap the vial and mix the two layers by inverting the vial several times. Be careful not to shake the vial as an emulsion (layer of bubbles between the two layers) can be created thus increasing the separation time.

9. Allow the two layers to separate. Transfer the upper organic solvent layer into a clean reaction vial using a pipette. This layer will contain ethyl ether and cinnamaldehyde.

10. Repeat steps 10 and 11. Combine the ethyl ether solutions from the two extractions into one reaction vial.

11. Dry the cinnamaldehyde solution with anhydrous magnesium sulfate. Add only a small amount (~2-3 mm on the bottom of the vial). If the magnesium sulfate is clumped up, add a little more until the magnesium sulfate stops clumping. Transfer the ethyl ether solution containing cinnamaldehyde to a clean, tarred evaporating dish using a pipette. Rinse the magnesium sulfate with 2-3 mL of clean ethyl ether and combine with the cinnamaldehyde solution in the evaporating dish. Record the mass of evaporating dish and evaporating dish /cinnamaldehyde solution.

12. Place the evaporating dish in a fume hood for 5-10 minutes allowing the ethyl ether to evaporate. Small droplets of cinnamaldehyde will remain after all the ethyl ether has evaporated. Ethyl ether has a low boiling point (40-70 ^{o}C). Cinnamaldehyde has a boiling point of 248 ^{o}C.

13. Smell the evaporating dish after all the ethyl ether has evaporated. The oily droplets will have a strong cinnamon smell. Mass the Petri Dish/cinnamaldehyde. Record

14. Test for the presence of an aldehyde by the Tollens (silver reduction) test. Place 1 mL of 10% silver nitrate and 1 mL of 10% sodium hydroxide in a small reaction vial. Add 6 M ammonium hydroxide drop wise until all the silver is dissolved. Add 1-2 drops of cinnamaldehyde to the Tollens reagent; seal the vial with a Teflon cap and shake for a minute. A positive test is the formation of a silver mirror in the vial after about 5 minutes. If no reaction occurs, warm the vial with hot water for a few minutes.

Data:

1. Initial Masses
   a. Mass of vial _____ b. Mass of vial/cinnamon _____ c. mass of cinnamon _____

2. Smell of cinnamon/water solution:

3. Final Masses
   a. Mass of Petri _____ b. Mass of Petri/cinnCHO _____ c. Mass of cinnCHO _____

4. Smell cinnamaldehyde: Compare to #2.

Calculations:

1. Calculate the percent yield of cinnamaldehyde. (Mass of cinnamaldehyde/Mass of cinnamon)

Questions:

1. Evaluate the percent yield of cinnamaldehyde extracted and explain why these chemical companies choose to synthesize these compounds instead of isolate them from natural sources.

2. Research some other synthesized flavors that are using the natural product. Be sure to include names and structures.