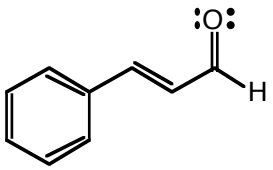
Isolation of Cinnamaldehyde from Cinnamon

Introduction

In this experiment you will isolate cinnamaldehyde from cinnamon. Commercial cinnamon consists of dried, ground bark from the cinnamon tree, and contains about 2% cinnamaldehyde, which is responsible for its distinct flavor and odor.

cinnamaldehyde 

The isolation will be accomplished by steam distillation. This means that the solid cinnamon will be boiled in water, and the steam will be condensed and collected. Since cinnamaldehyde is soluble in steam (but not in water), it will be carried up with the distillate and form a finely distributed emulsion, which will appear milky upon cooling. Many other essential oils can be isolated in this way – anisole from anise, camphene from nutmeg, carvone from caraway and spearmint, cuminaldehyde from cumin, eugenol from cloves, safrole from sassafras, and limonene from citrus peel.

Please read "Purifying a Liquid by Distillation" to learn how an ordinary distillation is run. Also read "Extracting and Washing," "Drying an Organic Solvent," and "Evaporating an Organic Solvent" before coming to lab. Write an introduction in your lab manual.

Procedure

Steam distillation of the cinnamon:

- Obtain a 100 ml Erlenmeyer flask with a 14/20 ground glass joint from the instructor.
- Add 15 ml of distilled water, 2 drops of Triton X-100 (a surfactant which reduces foaming), 2.0 g of cinnamon, and a long stir bar.
- Attach a Hickman still to the flask, then top it with a reflux condenser. Attach the reflux condenser to the water hoses and turn on the water. Insulate the top of the flask below the neck of the still with aluminum foil.
- Turn on the stirrer and begin to heat the cinnamon mixture slowly until it begins to boil.
- If it foams up into the still, you are heating too quickly – if the foam gets into the lip of the still, you'll have to take it off and clean it before continuing.
- Remove the distillate with a pipet as it collects and place it in a beaker or flask.

- If, after collecting some distillate, the still begins to look dry, add up to 1 ml of water (no more!). Try not to bake the cinnamon onto the glassware, as it is hard to clean out.

- You should collect about 5 ml of distillate; once it no longer turns milky on cooling, most of the cinnamaldehyde has been removed.

Isolation of the cinnamaldehyde from the distillate:

- Place a sep funnel on the clamp and put a beaker underneath it. After making sure the stopcock is closed, transfer the distillate to the sep funnel.

- Extract it by adding about 5-10 ml of dichloromethane, shaking, allowing it to separate, and draining off the dichloromethane.

- Repeat two more times, combining all of the dichloromethane layers that you drain off. Don't throw away anything until you are sure you have what you want!

- Dry the dichloromethane solution by adding sodium sulfate until it is free flowing.

- Transfer the solution to a tared (preweighed) round bottom flask and rinse the solid sodium sulfate with a little more dichloromethane. Evaporate the solution on the rotovap.

- Observe the product that you have obtained and record your observation. Authentic cinnamaldehyde is a clear, slightly yellow liquid with a strong odor of cinnamon.

- When the flask is cool, obtain the mass of the cinnamon oil that you have extracted. Calculate the % recovery of cinnamaldehyde.

- Discuss the odor, appearance, mass, and percent recovery of the cinnamaldehyde in your conclusion.

<u>Questions</u>

1) Cinnamaldehyde cannot be distilled directly because it decomposes before it reaches a boiling point (about 248^{o} C). How does steam distillation avoid this difficulty?

2) What would happen if you attached the reflux condenser to the flask first, then put the Hickman still on top?

3) Why does the distillate appear milky?

4) What would happen if you evaporated the dichloromethane solution without drying it first?

5) Why would you never expect to get a 100% recovery of cinnamaldehyde?