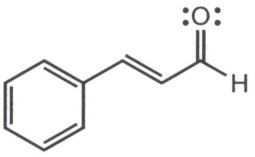
Isolation of Cinnamaldehyde

Introduction

In this lab, we will be separating cinnamaldehyde from commercial cinnamon, which comes from the bark of cinnamon trees. The cinnamaldehyde should only be about 2% of the original mass. We will use steam distillation to remove the cinnamaldehyde compound, because it is soluble in steam. We will then remove it from the water with dichloromethane, then dry and evaporate it to get the pure compound.

structure of cinnamaldehyde: 

Procedures	Observations
Set up steam distillation by putting 15 ml of distilled water, about 2.0 g of cinnamon, and 2 drops of Triton X-100 (to reduce foaming) into a 100 ml Erlenmeyer flask with a 14/20 ground glass joint on top. Aded a Hickman still on top, then a reflux condenser, and secured it with a clamp. I hooked up the tubing (into the bottom, out of the top), and folded some Al foil into a teepee and insulated the flask up to the lip of the still to help keep the heat in.	- actual mass of cinnamon: 2.021 g
Began heating slowly (starting on "3") to prevent too much foaming. Slowly heated until it began to boil (gradually moved up to "5").	- some foaming occurred, but it didn't get into the still - observed steam condensing into small droplets, then falling down into the lip of the still
Removed the distillate from the still as it collected, using a pipet. Put it in a small beaker. Watched for the flask to get dry, but it didn't seem to have a problem.	- distillate appeared milky as it cooled – this is because tiny droplets of cinnamaldehyde are coming out of solution, so this means cinnamaldehyde is present (it's working!) - also, strong cinnamon smell
Continued collecting until the distillate no longer appeared milky on cooling.	- collected about 6 ml
To remove the cinnamaldehyde from the water, set up a sep funnel on a clamp with a beaker underneath, and added the water/cinnamaldehyde mixture to it (checking the stopcock first). Added about 5 ml of dichloromethane, shook it, vented it, and then let it settle. Drained off dichloromethane into a beaker. Put in more dichloromethane, and repeated two more times.	- dichloromethane went to the bottom, water layer on top - water layer looked less milky after shaking (this means the cinnamaldehyde is being removed)

Checked for visible water droplets. Poured the solution into a clean beaker, leaving them behind.	- saw some drops of water around the bottom of the flask
Removed water from the dichloromethane solution by adding sodium sulfate. Stirred, added more, and watched for it to be free-flowing.	- some clumps formed, showing that water was being removed, then some grains were moving by themselves, showing that it is dry
Weighed a 50 ml round bottom flask. Poured dichloromethane solution into it, leaving the sodium sulfate behind. Rinsed the sodium sulfate with a few more ml of dichloromethane, and added it to the flask. Evaporated the solution on the rotovap.	- mass of empty flask: 50.365 g - evaporated easily, since bp = 34°C
Observed odor and appearance of product.	- clear, oily liquid; strong smell of cinnamon
Dried flask, let cool, and weighed. Calculated mass of cinnamaldehyde, then % recovery.	- mass of flask and cinnamaldehyde: 50.341 g - mass of cinnamaldehyde: 24 mg - 0.024 g / 2.021 g = 2.01 % recovery
Put water layer in the inorganic waste jug, sodium sulfate in the solid inorganic waste jar, and original cinnamon layer down the sink	

Conclusion

In this experiment, I successfully isolated cinnamaldehyde from commercial cinnamon powder. From 2.021 g of cinnamon, I obtained 24 mg of cinnamaldehyde, which is a 2.01% recovery. This is a reasonable amount, since there is usually about 2% cinnamaldehyde in cinnamon. The final product was a clear, oily liquid with a strong cinnamon smell.