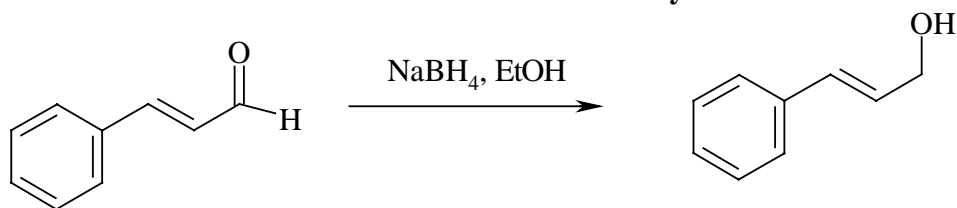


## The Reduction of Cinnamaldehyde



Until recently, most flavors and fragrances came from natural sources such as flowers, fruit etc. As chemistry has progressed, scientists have been able to synthesize these natural products. Trans-cinnamaldehyde is a major component of cinnamon. Our olfactory senses respond differently to different functional groups. In this lab we will reduce cinnamaldehyde to cinnamyl alcohol using sodium borohydride. The cinnamyl alcohol should smell a little like a hyacinth. Be sure to prepare your notebook to take data. Look up the FW, Mp or Bp of all the various compounds.

### Procedure:

1. In a 50 ml beaker, place 10 ml of 95% ethanol. Add a stirring bar and clamp a thermometer so that you can read the temperature of the solution. Begin stirring the solution.
2. Add 0.3 g of NaBH<sub>4</sub>.
3. Dissolve 2.5 ml of cinnamaldehyde in 4 ml of 95% ethanol. You may wish to use a dropping funnel.
4. Slowly add the solution making sure that the temperature does not exceed 60° C. After addition is complete, stir the reaction for an additional 10 minutes. Remove a tiny amount in a TLC spotter and do a TLC. If all the starting material is gone, very cautiously add 1 ml of 10% HCl and stir.
5. Remove the ethanol by heating the reaction IN THE HOOD until a second layer appears.
6. Cool the mixture and add 10 ml of ether and pour onto a separatory funnel. Rinse the beaker with a little ether to remove all the product.
7. Add 10 ml of cold water, shake and remove the water. Add 10 ml of saturated bisulfite and remove the bisulfite. Add 10 ml of cold water, shake and remove the water. These are called washes.
8. Dry the ether solution with anhydrous Na<sub>2</sub>SO<sub>4</sub>.
9. Decant the ether solution or filter the solution into a 50 ml round bottom flask. Remove the ether by vacuum evaporation. The product should be a solid. Check the smell of the product and compare it to the starting material.
10. Save your product.

**Post Lab Questions:**

1. If cinnamic acid is present as an impurity, will it be reduced by sodium borohydride?
2. Why is sodium borohydride caustic?
3. What form would the cinnamic acid impurity take after the borohydride reduction step?
4. Which reagent is used in excess in this reaction? Why?
5. What is the function of the hydrochloric acid in step 4?
6. What gas is formed in step 4?
7. Please interpret the NMR spectrum shown below.
  1. Draw the structure to show all hydrogens.
  2. Label the hydrogens a, b, c... Use the same label for equivalent hydrogens.
  3. Fill out the usual table
8. Please describe the changes you would expect in the IR from the starting material to the product.

