



Experiment 2

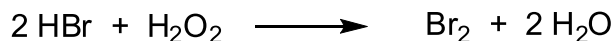
Reaction of *trans*-Stilbene with Hydrobromic Acid and Hydrogen Peroxide

Chemistry 251
Fall 2012

Background

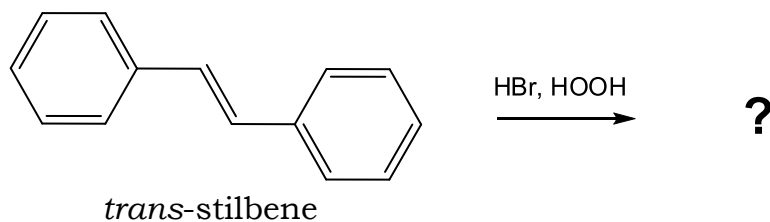
This semester you will learn many electrophilic addition reactions to alkenes, including hydrohalogenation, hydration, halohydrin, and halogenation as well as the radical addition of HBr to an alkene. In this lab, your task is to figure out what actually happens under a given set of conditions.

When hydrobromic acid reacts with hydrogen peroxide, molecular bromine and water are produced.¹



In theory, this leads to several different possible reactions with *trans*-stilbene...

- Electrophilic addition of HBr
- Electrophilic addition of Br₂
- Radical addition of HBr
- Electrophilic addition resulting in halohydrin formation



Prior to lab, you should write each reaction in your notebook and predict the possible products. (See Bruice, sections 4.1, 4.8, and 12.7)

You will perform the reaction according to the following procedure. You will use melting point to determine which product you synthesized.

¹ McKenzie, L.C.; Huffman, L.M.; Hutchison, J.E. *J. Chem. Ed.* **2005**, *82*, 306-310.

Experimental Procedure

SAFETY PRECAUTIONS: Care must be taken when using concentrated acid and/or 30% hydrogen peroxide. Both are corrosive, can cause eye and skin burns, and are harmful if inhaled. Be careful to avoid contact with skin and refrain from breathing these compounds. Neutralize all excess acid in the provided containers, and clean up all spills immediately. Acid will make holes in your clothes (and your skin), so try not to spill any. Ethanol is flammable so use caution.

1. Prepare a 100 mL round-bottom flask with a stir bar, and prepare a 90–100 °C water bath.
2. Measure out 0.6 g stilbene, and add it to the flask with 20 mL of ethanol. Fit with a reflux condenser and heat the reaction with stirring. Allow the solids to dissolve. Add a little more ethanol if everything doesn't go into solution.
3. Once dissolved, slowly add 1.0 mL of HBr (about 2.5 equivalents), and let the solution heat and stir. The precipitate caused by the addition of acid should go back into solution, but it may not. Continue even if it does not all go back in.
4. Measure out 1.0 mL of 30% hydrogen peroxide (also about 2.5 equivalents) and add it drop wise to the reaction. The color should change from clear and colorless (or cloudy white if the precipitate did not go back in) to dark golden yellow.
5. Let the reaction reflux and stir for about 20 minutes or until the color disappears and the mixture becomes a cloudy white.
6. Remove the reaction from the heat and let it cool. Once at room temperature, neutralize (pH 5 to 7) the solution with a concentrated NaHCO_3 solution. It may only take a little, depending on how much excess acid you have. Check pH with pH paper.
7. Once neutralized, put the flask on ice to further cool it and cause more crystals to precipitate. Collect the crystals by vacuum filtration, rinsing with very cold water and a little bit of very cold ethanol. Let air flow over the product to help dry it. Record your yield and product melting point.

NOTE: If you are unable to determine the product from your melting point, it may be that your product is too impure. You may recrystallize from ethyl acetate if necessary.

