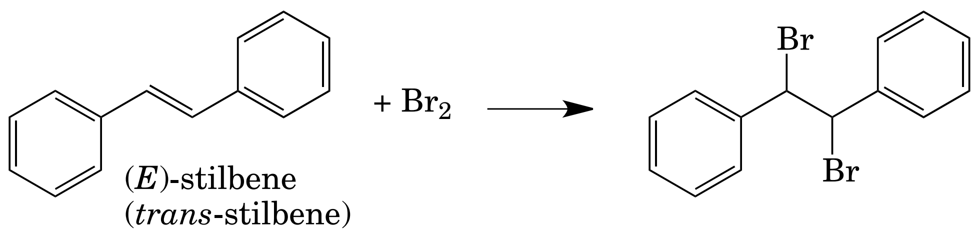
Stilbene Bromination

Reactions that incorporate halogens into compounds are widely used in organic chemistry. In the past, most of the reaction conditions employed toxic halogenated solvents, and have required addition of molecular bromine (Br_2), which is also toxic. The process that you will be running in this experiment uses ethanol, which is less toxic than most halogenated solvents, and generates the Br_2 within the reaction. It does, however, use concentrated hydrobromic acid and 30% hydrogen peroxide, both of which are potentially hazardous reagents.

The reaction shown below generates the Br_2 that will react with the stilbene:

$$2\ HBr + H_2O_2 \longrightarrow Br_2 + 2\ H_2O$$

Once the Br_2 is formed, it can react with stilbene:

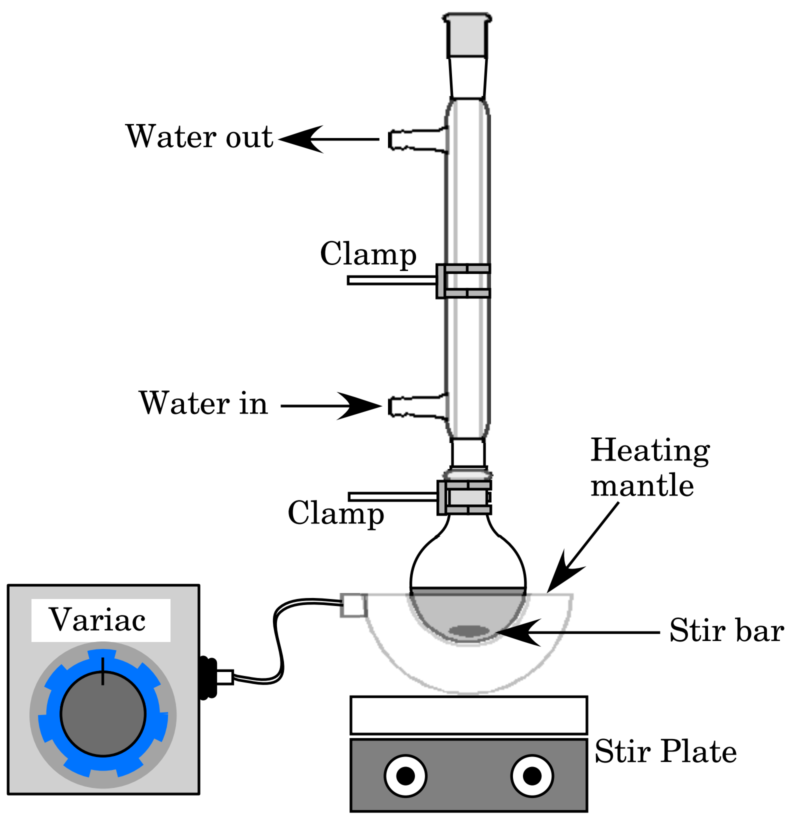


Procedure

Place 10 mL of ethanol, 0.5 g of stilbene, and a stir bar into a 100 mL round-bottomed flask. Place the flask in an appropriately sized heating mantle on a stir plate, and clamp the flask. Attach and clamp a condenser with hoses for water flow (the water flow rate should be low).

Plug the heating mantle into the variac, and turn on the power on setting 70. Heat the ethanol/stilbene mixture while stirring until the stilbene is mostly or completely dissolved.

Carefully add 1.2 mL concentrated hydrobromic acid and continue heating and



stirring. It is normal for some of the stilbene to precipitate; the precipitated stilbene should redissolve during the reaction. Add 0.8 mL 30% hydrogen peroxide in dropwise fashion. The solution should become dark yellow as the hydrogen peroxide is added. Continue refluxing until the reaction mixture is a cloudy white in appearance (about 20 minutes).

When the reaction is complete, switch off the variac, and carefully remove the stir plate from under the flask (if your apparatus is clamped properly, the flask and condenser should remain suspended, while the heating mantle will fall to the hood floor. Move the heating mantle (carefully, because it will be hot) to the laboratory bench to avoid melting the hood floor.

Once the mixture has cooled, add 10% $NaHCO_3$ to the mixture until bubbles no longer form. Cool the mixture on ice to precipitate more of the product, and then collect the product by vacuum filtration. Allow the product to dry in your drawer before measuring melting point and mass.

Report Guidelines:
Submit one neatly typed report per pair (this sheet does not need to be included)

1. The report should include the grading rubric as cover sheet, with the appropriate information filled in. (5 points)

2. (5 points) Describe the experimental procedure that you followed in a manner consistent with the experimental section of a chemistry research paper. The description should be a technical, accurate report of what you did, written in past tense. Avoid the temptation to be overly verbose, but be sure to include every detail necessary for a student with your level of training to execute the lab. Include observations on all significant changes during the course of the reaction. Finally, draw a curved arrow formalism reaction mechanism for the addition of Br_2 to stilbene.
3. (5 points) Report the melting temperature range of the product. The pure product has a melting point of 241°C. Based on the melting temperature of your crude product, judge the relative purity of your compound. If the melting point is significantly different, propose a reasonable explanation for the discrepancy. You should also determine the melting point for the stilbene starting material.
4. (5 points) Using the masses of product, calculate the percent yield of the reaction. Show your calculations (this can be neatly written and not typed).
5. (5 points) Attach a hard copy of each lab partner's lab notebook entry to the assignment.

Reference: Adapted from Doxsee, K.M. and Hutchison, J.E. *Green Organic Chemistry – Strategies, Tools, and Laboratory Experiments*, Thompson Brooks/Cole, 2004, pp. 125-128.