Chem 51LB: EXPERIMENT 2
STEREOCHEMISTRY OF BROMINE ADDITION TO TRANS-CINNAMIC ACID

REACTION: Bromination of an Alkene
TECHNIQUES: Microscale Reflux, Recrystallization, Melting Point

In this experiment, we will learn how experimental results can be used to propose a reaction mechanism. Specifically, we will study the mechanism of addition to an alkene using trans-cinnamic acid and molecular bromine.

READING ASSIGNMENT:
- Review recrystallization technique from last week.
- Supplementary information in Janice Gorzynski Smith (2nd ed), Chapter 10, pp. 381-384.

PRE-LAB ASSIGNMENT:
- Follow the instructions in the “51LB Report Guidelines” document located on the course website to prepare your pre-lab.
- Complete the online Sapling pre-lab questions.

IMPORTANT SAFETY INFORMATION
Bromine is very corrosive and causes serious burns. Its vapors are extremely toxic and irritating to the eyes, mucous membranes, and respiratory tract. Keep bromine in the fume hood. Avoid inhalation and contact with skin and eyes.

Cyclohexene is flammable and an irritant. Its vapors are irritating to the eyes, mucous membranes, and respiratory tract. Keep cyclohexene in the fume hood. Avoid inhalation and contact with skin and eyes.

Dichloromethane is toxic, an irritant, absorbed through the skin, and harmful if swallowed. Wear gloves and wash your hands thoroughly after handling it. Avoid contact with skin and eyes. Dichloromethane is a suspected carcinogen when inhaled in large quantities. Keep it in the fume hood.

trans-Cinnamic acid is a mild irritant. Avoid skin contact and wash your hands after handling it.

EXPERIMENTAL NOTES:
REACTION PROCEDURE:
In a 10 mL round-bottomed flask, add 100 mg of trans-cinnamic acid followed by 0.7 mL dichloromethane. Add a few boiling chips to the flask and equip it with a water-cooled condenser. (Note: make sure the clamp is holding the neck of the flask and not the condenser!) Once you are ready with your reaction set up, ask the TA to come over and add 0.35 mL of 10% bromine in dichloromethane solution to your flask. **You are not allowed to handle the bromine!** Lower your apparatus into a warm water bath (Use a 250 mL beaker filled up with water and heat it to 40-50 °C). Heat the reaction to reflux for 20 min.

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NOTES:
If the temperature of the bath gets too hot, add some cold water to avoid boiling off all of your solvent. (What’s the boiling point of dichloromethane?) If you do boil off too much of your solvent, you can add additional solvent as necessary at any time. If the reaction turns from brown to colorless during the 20 min of reflux, ask your TA to add additional bromine to your reaction.

WORK-UP:
Allow the reaction to cool to room temperature. Add cyclohexene dropwise until the color ceases to fade further. The solution may not go completely colorless, but it should go to a faint orange. Add only the minimum of cyclohexene you need.

Safety notice: Some students are particularly sensitive to the odor of cyclohexene and may develop headaches or dizziness. Ensure that cyclohexene is used exclusively in the fume hood and in the smallest necessary amounts.

Cool the reaction flask in an ice bath to ensure that all of your product has crashed out of solution. Filter the reaction mixture using a Hirsch funnel and collect the solid. Please note that Hirsch funnel frits are NOT DISPOSABLE and should be reused. Hirsch funnel filter paper should be placed on top of the frit prior to filtering and is disposable.

Recrystallize the solid by dissolving it in a minimum amount of hot (nearly boiling) ethanol. Add hot water slowly until a slight cloudiness persists after stirring. Add a few drops of hot ethanol just until all cloudiness disappears. Proceed with the recrystallization as you normally would. After filtering and drying the solid, obtain a melting range for the purified product.