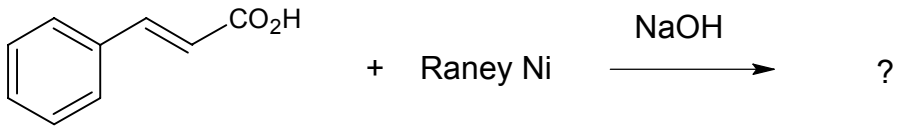
Solving a Mystery Reaction

Introduction

In this experiment, you will be deducing the structure of the product of a reaction not covered in your textbook. *trans*-Cinnamic acid will be dissolved in aqueous sodium hydroxide and then treated with a nickel-aluminum alloy called Raney Nickel. Raney nickel is composed of approximately equal amounts of nickel and aluminum and has a formula that is approximately $NiAl_2$. Learn more about Raney nickel at http://en.wikipedia.org/wiki/Raney's_nickel



trans-Cinnamic acid contains two functional groups, a carboxylic acid and an alkene. Carboxylic acids undergo acid base reactions to form water soluble carboxylate salts with bases such as sodium hydroxide. In your laboratory notebook, write the equation for the reaction of *trans*-cinnamic acid with sodium hydroxide. Carboxylate salts can be converted back to the carboxylic acid by acidification with acids such as HCl or H_2SO_4.

IMPORTANT SAFETY INFORMATION:

Treatment of Raney Nickel with sodium hydroxide makes it very reactive. **You must keep it wet at all times. If you allow it to become dry, it may spontaneously ignite!** When you are done with your Raney nickel, put it in the Raney nickel waste container in the hood. This container contains water to keep the Raney nickel from catching fire.

Procedure

The experimental procedure is described for the use of 1.0 g of *trans*-cinnamic acid. Weigh out 0.5 to 1.0 g of *trans*-cinnamic acid, and adjust the amounts of the other reagents to match your starting amount of *trans*-cinnamic acid.

Place 1.0 g of *trans*-cinnamic acid and 50 mL of 5% NaOH into a 250-mL Erlenmeyer flask. Heat the mixture to 90 ^{o}C on a hot plate to dissolve the solid. When the *trans*-cinnamic acid is dissolved, remove the flask from the hot plate. Add 3.0 g of Raney nickel powder in small portions over a period of 15 minutes to the solution. Swirl the flask while adding the powder to the center of the swirling liquid. Note in your lab notebook what happens as you add the powder. After adding all of the Raney nickel, return the flask to the hot plate and heat at 90-95 ^{o}C for 30 minutes. With your Sharpie marking pen, draw a line on the Erlenmeyer flask to indicate the initial level of the liquid.

Heat the mixture with frequent stirring for 30 minutes. During this period, add hot water to maintain the level of the liquid.

Filter the solution by vacuum filtration. Add 4-5 mL of hot 5% NaOH to wash the Erlenmeyer flask. Pour these washings through the black residue in Büchner funnel. As soon as the filtration is complete, using water as a transfer agent, collect the black nickel material into a beaker. You ***must*** keep this residue wet! Dispose of this material in the waste container in the hood.

Cool the filtrate remaining in the filter flask to room temperature in an ice bath contained in one of the rectangular plastic containers (this container can hold two filter flasks simultaneously). Slowly with stirring, add the cooled mixture to a beaker containing 30 mL of concentrated HCl. Add the mixture at a rate that allows the temperature to not exceed 85 ^{o}C. When the addition is complete, cool the mixture in an ice bath. Collect the white precipitate by vacuum filtration. Wash the precipitate with 10-10 mL of cold water.

Recrystallize your product with hot water, allow the product to dry and determine its melting point.

Determining Structure

Since there are two functional groups in the starting *trans*-cinnamic acid, it is important to determine whether either or both of them react with the reagents used in the procedure. To make this determination, you will carry out three simple tests on your product.

(1) Test the solubility of the product in 5% NaOH at room temperature or with heating.
(2) Test the product with cold $KMnO_4$ (Baeyer test).
(3) Test the product with bromine in CCl_4.

What do these tests tell you about the functional group or groups in your product?

Since the product is a solid, the melting point can be used for identification purposes. Compare your melting point with the melting points of some known potential products.

Compound	**Mp (^{o}C)**
Trans-cinnamic acid	133-134
Cinnamyl alcohol	33-35
Benzoic acid	122-123
3-phenylpropanoic acid	47-49
2-hydroxy-3-phenylpropanoic acid	96-97
3-hydroxy-3-phenylpropanoic acid	92-93
Phenylpyruvic acid	154-155
Benzoylacetic acid	103-104
1-phenyl-1,2-ethanediol	66-68

The Lab Notebook

You need to include the melting point of your product and your conclusions based on the table above along with the results and conclusions from the solubility and chemical tests. You need to assign a structure (draw it out) to your product and present your arguments that support it.