

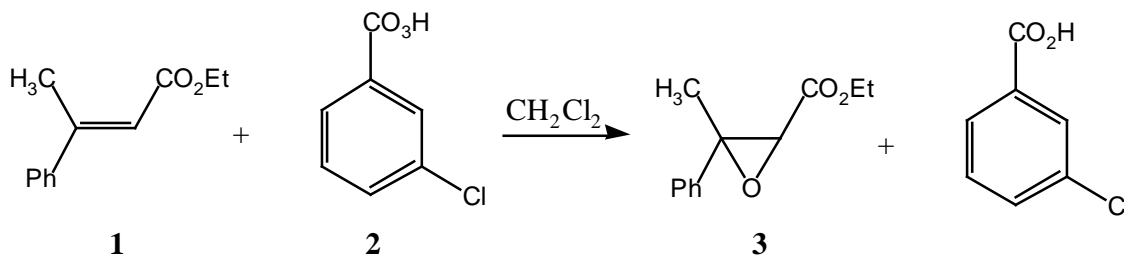
## CH 241 EXPERIMENT #7

### WEEKS OF NOVEMBER 12 and 26, 2001 EPOXIDATION OF AN ALKENE: THE SYNTHESIS OF AN ARTIFICIAL "STRAWBERRY" FLAVORING AGENT

(This experiment is based upon the adaptation of literature procedures<sup>1,2</sup> by the following Colby students: Gayle Pageau '02, Rodwell Mabaera '02, Kathryn Kosuda '02, Tamara Sebelius '02, Ali Ghaffari '02, and Ken Kearns '01)

#### Introduction

In class you will see that alkenes undergo a wide variety of addition reactions. In this lab you will perform one such reaction by epoxidizing ethyl *trans*- $\beta$ -methylcinnamate (**1**) with *m*-chloroperbenzoic acid, *m*-CPBA (**2**). The product of this reaction, epoxide (**3**), is a popular food flavoring agent that is used to impart the flavor of strawberries. The reaction is shown below.



After carrying out the reaction and purifying the product, you will need to characterize the purified epoxide by  $^1\text{H}$  NMR spectroscopy.

#### Experimental

##### Week 1: Epoxidation

In a clean and dry 20 mL scintillation vial equipped with a magnetic stir bar, weigh out 0.57 g of 70-75% *m*-chloroperbenzoic acid. Add 2.5 mL of dichloromethane and gently stir the solution. Meanwhile, prepare a solution of 0.25 g of ethyl *trans*- $\beta$ -methylcinnamate dissolved in 1.25 mL of dichloromethane. Using a pipet, slowly add this solution to the vial while stirring. Allow the mixture to stir until the *m*-chloroperbenzoic acid has completely dissolved. Remove the stir bar, label the cap of your vial with your name and lab day, and put the reaction mixture into the cold room until your next lab period.

##### Week 2: Purification and Characterization of Strawberry Aldehyde

Decant your reaction mixture into a 125 mL separatory funnel. Rinse the residue with an additional 5 mL of dichloromethane and add the rinse solution into the same separatory funnel. Wash the organic layer with 10 mL portions of 10% aqueous sodium sulfite until the washings give a negative test with starch-iodide paper. This ensures that all excess peracid has been destroyed. Then wash the organic layer with two 10 mL portions of 5% aqueous sodium bicarbonate to remove benzoic acid. Dry the organic layer over anhydrous sodium sulfate and filter through a Pasteur pipet packed with silica gel into a clean, dry, and tared round bottom

flask. Remove the solvent using a rotary evaporator and reweigh the flask to get a crude yield. Use about 15-20 mg of the product to prepare an NMR sample in  $\text{CDCl}_3$  and acquire a  $^1\text{H}$  NMR spectrum.

### Prelab Assignment

Before coming to lab during the week of November 26, please do the following.

- (a) Read section 9.3a in your text.
- (b) The product of your reaction, compound **3**, is commercially known as "strawberry aldehyde." Explain what is odd about this name.
- (c) Predict the splitting pattern you expect to see for the ethyl group in the  $^1\text{H}$  NMR spectrum of the product.

### Report

- (1) Write a detailed experimental procedure for the epoxidation reaction that you carried out and attach the  $^1\text{H}$  NMR spectrum of the product.
- (2) Write a mechanism for the epoxidation reaction and explain how many isomers of the epoxide might be expected in this reaction.
- (3) Assign the correct configuration to the stereogenic center(s) in the epoxide(s) expected from this reaction.
- (4) Analyze the  $^1\text{H}$  NMR spectrum of the epoxide paying particular attention to the splitting pattern observed for the methylene signal.