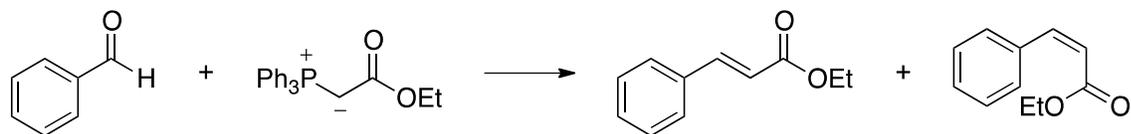


Wittig Reaction

Question: Does the stabilized Wittig reaction to form Ethyl Cinnamate lead primarily to the *cis* or *trans* product?

Background: Formation of C-C and C=C bonds is essential to organic synthesis. A number of means have been developed. Today you will carry out the Wittig reaction using a stabilized ylide and analyze the product by NMR.

Procedure: Weigh 0.201 g of (carboxymethylene)triphenylphosphorane into a 3mL or 5mL conical vial and add 50 μ L of benzaldehyde. Stir/grind the mixture with a small spatula at room temperature for 15 minutes. At this point, the reaction should be complete. Add 3 mL of hexanes and stir for a few more minutes. The solid triphenylphosphine oxide byproduct will crash out of solution. Allow the solid to settle, and remove the liquid with a filter pipet. Place into a clean, tared container. Add a second 3 mL portion of hexanes to the reaction vessel, rinse the solids, collect the hexanes and combine with the other hexane extract.. Evaporate the combined hexane portions. If solid is observed during the evaporation, repeat the filtering process. Obtain a mass of the product and prepare a sample for NMR using CDCl_3 .



Your final product will be a mixture of the *cis* and *trans* alkenes. Using NMR analysis, your goal will be to determine the product distribution and the identity of the major product.

Results: In the results section, include a table of NMR data taken from your spectrum. Include all pertinent data necessary to determine major product identity and product distribution

Comments: What is an ylide? What is a stabilized ylide? Does the stabilized Wittig formation of ethyl cinnamate lead primarily to the *cis* or *trans* product? What data support this conclusion? What was the *cis/trans* product ratio of this reaction? What data support this conclusion? What was your overall yield? How could this reaction be improved in the future?

Lab 11 assignment: Turn in a hardcopy of your lab notebook (carbon copy or photocopy) and a hardcopy of your proton NMR.

- Due at the end of lab THIS WEEK
- 25 pts based on in-lab performance, completion of all sections, correctness of content