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Author(s): MELO 3D Project Team, 2012

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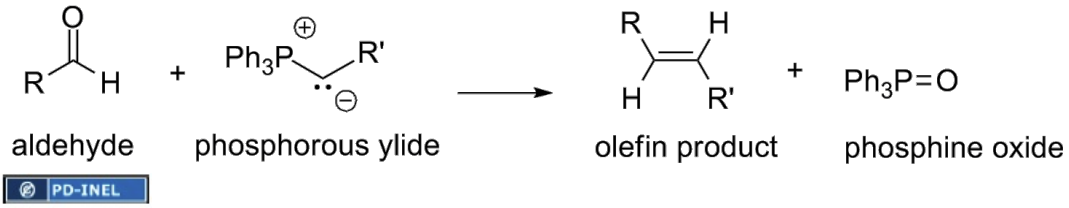
ch216sp12syllabus: Experiment 6

Experiment 6: The Wittig Reaction

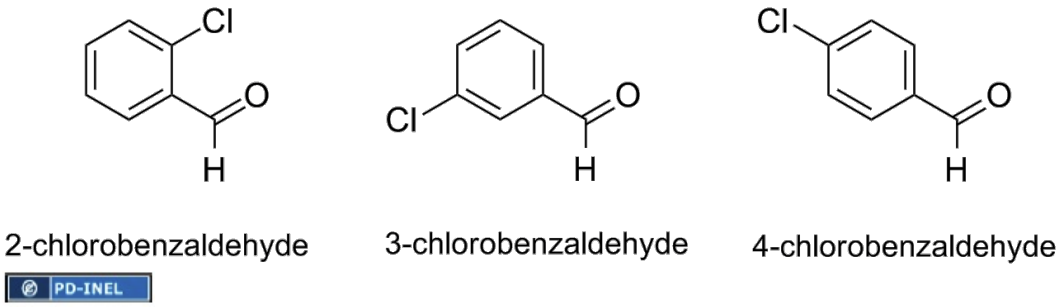
Introduction: The Wittig reaction is one of the most widely used methods for forming carbon-carbon double bonds, because it is easy to carry out and often gives high yields of pure product. It is named after its discoverer, German chemist Georg Wittig.[1] The reaction involves the addition of a phosphorus ylide to an aldehyde or ketone to form double bond with the elimination of phosphine oxide.

Reaction Scheme:



1. Wittig, G. *Pure & Appl. Chem.* **1964**, *9*, 245-254

Objective: Experiment 6 is a group experiment. You will work in a small group to carry out variations of the Wittig reaction and to answer the focus questions below. Each group member will perform the Wittig reaction with commercially available ylide (carbethoxymethylene)triphenylphosphorane and one of the three aldehydes (2-, 3-, or 4- chlorobenzaldehyde) below. Group members will work together to identify appropriate solvents for use in TLC and purification by microscale wet column chromatography. Be sure to complete the group design sheet.



Focus Questions (answer all):

1. Would you expect the position of the Cl group to affect the reactivity of the aldehyde or the outcome of the reaction, and how could you measure this using the equipment available?

2. The three products are constitutional isomers. Can you distinguish them using the available characterization methods in 216?) If so, what will these methods tell you?

Techniques: wet column chromatography, thin layer chromatography, and infrared spectroscopy.

Procedure: Dissolve chlorobenzaldehyde (50 mg) in dichloromethane (3 mL) in a dram vial equipped with a stir vane. Add 1.2 mol equivalents of the ylide (mol. wt. 348.38 g/mol) portion wise while stirring. Stir at room temperature for two hours while monitoring the reaction by TLC. When the reaction is complete evaporate the dichloromethane solvent with a stream of N_2 gas and dissolve the reaction mixture in 25% diethyl ether in hexanes (2-3 mL). Note the formation of a white precipitate, which is triphenylphosphine oxide. Transfer the solution to a clean vial and evaporate the majority of the solvent. Purify the crude product using a microscale wet column.

Post lab Questions

1. Why does triphenylphosphine oxide precipitate from a mixture of 25% diethyl ether in hexanes, but the olefin product remains dissolved?

2. Is more than one product obtained in any of the three reactions? If so what is the major product?

3. How do the three olefin products (from 2-,3-, and 4- chlorobenzaldehyde) compare by TLC, m.p., and Infrared spectroscopy?