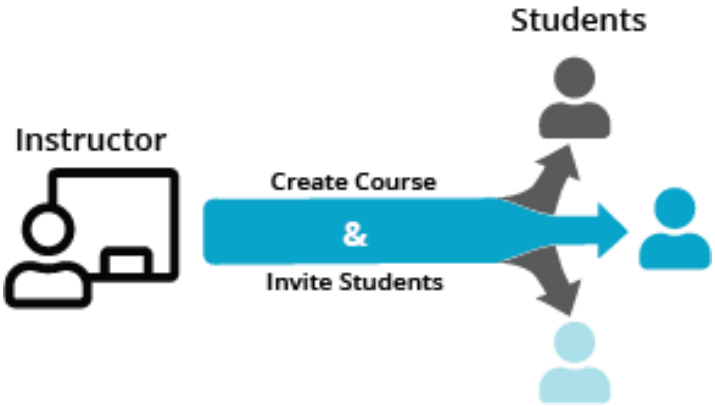
labarchives
Better Science

LabArchives is invested in supporting digital learning and Open Educational Resources (OER). Hundreds of labs and protocols are available through Lab Builder, our open source library.

To find out more and to gain free access to our complete library of content, please visit our website to sign up for an account.

Lab Builder, by LabArchives, also offers OpenStax textbooks at no cost inside of your LabArchives account. Build a complete course using the LabArchives notebook platform, course management tools, and our open digital library. Please contact Support with any questions on how begin implementation in to your course.



To stay up to date on all things LabArchives and read about our numerous success stories, please visit our Blog.

labarchives
Lab Builder

Wittig Reaction

Ginger Shultz
Assistant Professor of Chemistry
College of Literature, Science, and the Arts
University of Michigan
gshultz@umich.edu

Attribution

Instruction Level

- Undergraduate Lower Division

Topic

- Wet column chromatography
- Thin layer chromatography
- Infrared spectroscopy

Instruction Type

- Traditional

Timeline

- **Procedure:** 2 hours

Learning Objectives

- 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.

Introduction

Background

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

Pre-Lab Assignment

1. *Purpose Statement:* In one or two sentences, specifically describe the purpose of the day's experiment/lab work. What are you trying to learn or achieve, and how?

2. *Reagents:* List out all of the reagents you will be using in this experiment. For each of the reagents list their basic physical properties as well as any specific safety or handling precautions. This information can be found on a SDS.

3. 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?

4. The three products are constitutional isomers. Can you distinguish them using the available characterization methods you have used so far in this course? If so, what will these methods tell you?

***Before lab discuss your focus question with your small group and answer the following questions:*

Group Experimental Design

1. What is your hypothesis?
2. What specific experiments and/or reactions will you run to test your hypothesis?
3. How will you measure the results of your experiment(s)and what will these results tell you? (i.e. how will you know your hypothesis is correct or incorrect?)
4. How will the work be divided in the group and how will you ensure that the experiments are set up reproducibility and consistently between group members?
5. How will you ensure that it is a fair experiment and what are the dependent and independent variables?

Procedure

Safety

- General chemistry laboratory safety should be observed in this course.
- Personal Protective Equipment (PPE) including gloves, splash proof goggles, and protective apron must be worn at all times.
- Experiments should be carried out under a fume hood.

Protocol

Procedure

1. Dissolve chlorobenzaldehyde (50 mg) in dichloromethane (3 mL) in a dram vial equipped with a stirring vane.
2. Add 1.2 mol equivalents of the ylide (mol. wt. 348.38 g/mol) portion-wise while stirring.
3. Stir at room temperature for two hours while monitoring the reaction by TLC.
4. 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.
5. Transfer the solution to a clean vial and evaporate the majority of the solvent.
6. Purify the crude product using a microscale wet column.
7. Analyze the purified product using IR spectroscopy.

Results

Describe your observations of the IR spectra of the purified experimental product.

Post-Lab Assignment

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?