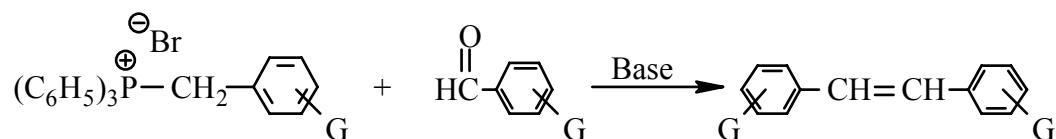


Unit 2M: Introduction to Organic Synthesis II
Preparation of Stilbene (Last update 09/13/04)

This assignment is due at the beginning of your recitation section one week before the day that you are scheduled to perform this experiment. It is in your best interests to complete these exercises as far in advance as practical. It is not in your best interests to wait until the day you have lab.

To receive a satisfactory grade your answers must be presented clearly. Looks count. Do your preliminary work on scrap paper. You can find data about the physical properties of the compounds in question in an Aldrich catalog, copies of which are available in rooms 152 and 363 of the Science building.

In the **Introduction** to this Unit, the Wittig reaction was described in general terms by the following equation:



The base that is used to deprotonate the phosphonium salt was not specified. Questions 1-3 involve deprotonation of benzyltriphenylphosphonium bromide with three different bases, lithium ethoxide, sodium hydride, and n-butyl lithium.

1. Write a balanced equation for the reaction of benzyltriphenylphosphonium bromide with lithium ethoxide, $\text{LiOCH}_2\text{CH}_3$.

2. Write a balanced equation for the reaction of benzyltriphenylphosphonium bromide with sodium hydride, NaH .

3. Write a balanced equation for the reaction of benzyltriphenylphosphonium bromide with n-butyl lithium, $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{Li}$.

4. In the deprotonation of benzyltriphenylphosphonium bromide with lithium ethoxide, the actual base that removes the proton from the carbon atom is the ethoxide ion; lithium ethoxide is technically the source of this base rather than the base itself. Furthermore, the oxygen atom is the basic atom in the ethoxide ion. The following table lists several bases that are commonly used in organic chemistry. Following the example give for lithium ethoxide, complete the table by drawing the structures of the actual bases that are produced from each of the sources listed. Show a lone pair of electrons and the charge on the basic atom.

Source of Base	Name	Actual Base
$\text{LiOCH}_2\text{CH}_3$	lithium ethoxide	$\text{:OCH}_2\text{CH}_3$
NaH	sodium hydride	
$\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{Li}$	n-butyl lithium	
NaNH_2	sodium amide	
$\text{LiN}[\text{CH}(\text{CH}_3)_2]_2$	lithium diisopropyl amide	
NaOH	sodium hydroxide	
KOH	potassium hydroxide	
NaOCH_3	sodium methoxide	
$\text{KOC}(\text{CH}_3)_3$	potassium t-butoxide	

5. What mass range, in milligrams, corresponds to 1.00 ± 0.05 mmol of benzyltriphenylphosphonium bromide. Show your work for 0.95 mmol

$$0.95 \text{ mmol} = \underline{\hspace{2cm}} \text{ mg}$$

$$1.05 \text{ mmol} = \underline{\hspace{2cm}} \text{ mg}$$

6. The procedure calls for 1.0 equivalents of benzaldehyde for every mmol of benzyltriphenylphosphonium bromide. How many mL of 0.25 M benzaldehyde solution should you use for 0.95 mmol of benzyltriphenylphosphonium bromide? Show your work.

7. What is the theoretical yield of triphenylphosphine oxide, $(\text{C}_6\text{H}_5)_3\text{P}=\text{O}$, that can be formed from 1.05 mmol of benzyltriphenylphosphonium bromide? Show your work. _____ mL

8. What is the theoretical yield of stilbene that can be formed from 1.05 mmol of _____ mg
benzyltriphenylphosphonium bromide? Show your work.

9. Assume that you started with 0.98 mmol of benzyltriphenylphosphonium bromide and _____ mg
that you isolated 47.3 mg of stilbene. What was your % yield? Show your work?

_____ %