

Post-Laboratory Report

Name _____

**Unit 2M: Introduction to Organic Synthesis II
Preparation of stilbene (Last updated 08/18/04)**

1. Write an equation for the reaction you performed:

2. Complete the following table. Record masses to the nearest mg. Record R_f values to two decimal places.

Compound	MW	mass, mg	mmol	R_f
benzaldehyde				
benzyltriphenylphosphonium bromide				
n-butyl lithium	----	-----		----
<i>trans</i> -stilbene				

3. What solvent system did you use for your TLC? For example, if you mixed 7 mL of hexanes with 3 mL of ethyl acetate, your answer would be 7/3 hexanes/ethyl acetate.

4. a. What was the theoretical yield of stilbene in your reaction? Show your work.

_____ mg

b. What was the per-cent yield of crude stilbene(s) in your reaction? Show your work.

_____ %

5. What was the melting point range of your crude stilbene? _____ °C to _____ °C

6. Complete the table provided on the next page. Refer to your lab manual for a listing of approximate frequencies for the vibrations listed. Use the spectrum of the sample of stilbene that you prepared for the measured frequencies. A discussion of characteristic group frequencies of arenes is available on pages 95-101 of the supplementary website data for Chapter 6 of your lab manual. Copies of this document are available in rooms 152 and 367 of the Science building.

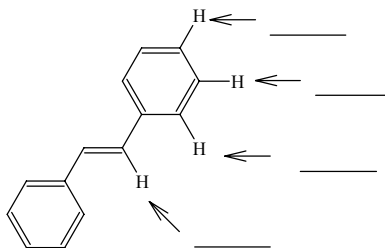
Staple your IR spectrum to this sheet. Make sure it is labeled with your name, the name of the compound, a structural drawing of the compound, a reference to the page in your notebook where you describe the synthesis of the compound, and the file name that you used to store the spectrum on the IR computer. Each of the peaks in the table below should be labeled on your spectrum.

Peak	Vibration	Approximate Frequency, cm^{-1}	Measured Frequency, cm^{-1}
1	vinyl C-H stretch		
2	vinyl C=C stretch		
3	arene C-H out-of-plane bending (5H)		
4	arene C=C ring stretch (ν_{8a})		
5	arene C=C ring stretch (ν_{8b})		
6	arene C=C ring stretch (ν_{19a})		
7	arene C=C ring stretch (ν_{19b})		
	arene C=C o-o-p ring deformation (ν_4)		

7. a. What was the GC retention time of your sample of stilbene? _____ minutes
- b. What was the m/z value of the base peak in your MS? _____

Staple your GC-MS Result File to this report. The mass spectrum must correspond to the retention time of the peak for stilbene in your gas chromatograph.

8. a. Staple your $^1\text{H-NMR}$ spectrum to this report.
- b. Enter the chemical shift of each of the H atoms indicated by the arrows in the following diagram. Note-You may use the ACD Labs H-NMR Predictor program to calculate the theoretical 60 MHz $^1\text{H-NMR}$ spectrum of *trans*-stilbene. That program is available on the computer that controls the 60 MHz spectrometer. The chemical shift values you enter here should be your experimental values.



9. a. Staple your ^{13}C -NMR spectrum to this report.
b. Enter the chemical shift of each of the C atoms indicated by the arrows in the following diagram. Note-You may use the C-NMR Predictor program to calculate the theoretical 40 MHz ^{13}C -NMR spectrum of *trans*-stilbene. That program is available on the computer that controls the 60 MHz spectrometer. The chemical shift values you enter here should be your experimental values.

