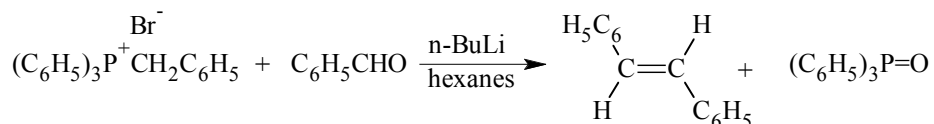


Preparation of *trans*-Stilbene

Materials	FW	Amount	mmol
benzaldehyde	106.1	5.8 mL (0.25M)	1.5
$\text{C}_6\text{H}_5\text{CH}_2\text{P}(\text{C}_6\text{H}_5)_3 \text{Br}$	447.3	0.6594 g	1.522
n-BuLi	1.6 M	0.90 mL	1.4

Reference: This procedure was based on the Unit 2 handout for CHY 252.

Procedure:

In a 25 mL round-bottomed flask, I stirred a suspension of 0.6594 g (1.522 mmol) of benzyltriphenylphosphonium bromide in 10 mL of hexanes for 10 minutes in a dry ice/acetone cold bath. I then injected 0.90 mL of 1.6 M n-butyl lithium (1.4 mmol) which caused the solution to turn bright orange indicating the formation of the ylide. After 30 minutes, I injected 5.8 mL of a 0.25 M solution of benzaldehyde (1.5 mmol) in hexanes into the flask. The mixture changed from bright orange to off-white. I capped the flask and set it aside for one week. I added about 5 mL of hexanes to the flask and filtered the mixture, rinsing the solid with small portions of hexanes. I evaporated the filtrate using a rotary evaporator. The residue weighed 0.137 g. A TLC of this crude product, using a 4/1 mixture of hexanes/methylene chloride as the eluent, showed three spots with R_f values of 0.52, 0.47 and 0.27. I spotted the above TLC plate with two controls dissolved in small portions of methylene chloride. The *E*-stilbene and triphenylphosphine oxide controls (both authentic samples from Sigma-Aldrich) and yielded spots at 0.51 and 0.30, respectively.

I recrystallized the crude material from boiling 95% ethanol. I recovered 0.050 g (20%) of white needles. The melting point of this material was 120-122 °C. I recorded the IR, ^1H NMR, and GC-MS spectra of this sample.

Analysis of Data:

The R_f values of 0.52, 0.47 and 0.27 for the crude product closely matched those of authentic samples of *E*-stilbene and triphenylphosphine oxide (R_f values of 0.51 and 0.30 in a 4/1 mixture of hexanes/methylene chloride as the eluent). I assign the spot with an R_f value of 0.47 to *cis*-stilbene. I repeated the TLC analysis on the recrystallized sample and obtained only one spot with an R_f value of 0.50, indicating that only *trans*-stilbene was present. The *cis*-stilbene in my crude product mixture was lost in the recrystallization procedure as I expected it to be since *cis*-stilbene is much more soluble in 95% ethanol than *trans*-stilbene. The melting point of my sample was slightly lower than the published value¹ of 122-124°C.

The IR spectrum of my product shown is in Figure 1. There were peaks at 3078 and 3058 cm^{-1} , which I assigned to aromatic C-H stretches. I also assigned a medium intensity peak at 3017 cm^{-1} to the C-H stretching of the *trans*-substituted double bond.

¹ The Aldrich Catalog, 2005-2006, p. 2149.

The four peaks I expected for arene C=C stretching, ν_{8a} , ν_{8ba} , ν_{19a} , and ν_{19b} , appeared at 1593, 1573, 1491, and 1450 cm^{-1} , respectively. These values compare well with those listed on pages 97-98 of the discussion entitled "Spectroscopic Identification of Organic Compounds by Infrared Spectroscopy"². I assigned a strong peak at 963 cm^{-1} to the aromatic C=C out-of-plane ring deformation and another strong band at 764 cm^{-1} to the C-H out-of-plane bending mode in a mono-substituted aromatic ring.

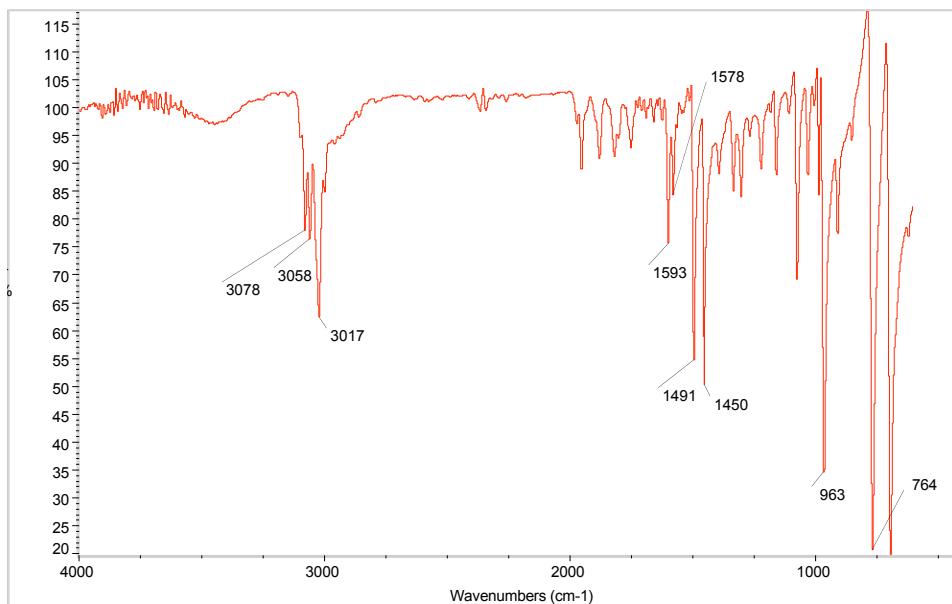


Figure 1. The IR Spectrum of *trans*-Stilbene.

I dissolved 30 mg of my purified stilbene in 700 μL of CDCl_3 . The ^1H NMR spectrum of this material showed a small peak near 6.5 ppm, a singlet at 7.01 ppm and a group of tightly spaced peaks between 7.14 and 7.43 ppm. I think the peak at 6.5 ppm is due to an impurity. I assigned the singlet at 7.01 ppm to the vinyl protons, and the multiplet between 7.14 and 7.43 ppm to the aromatic protons. According to the ACD/H NMR Predictor the chemical shift of the vinyl proton in *trans*-stilbene occurs at 7.10 ppm. The ratio of the aromatic to vinyl protons was 5.0/0.95, which is very close to the expected value of 5/1. The spectrum is reproduced in Figure 2.

² Website data for Chapter 6 of *Microscale Techniques for the Organic Laboratory*, 2 ed. by D. W. Mayo, R. M. Pike, and P. K. Trumper; 2001, John Wiley and Sons, NY. ISBN 0-471-24909-2

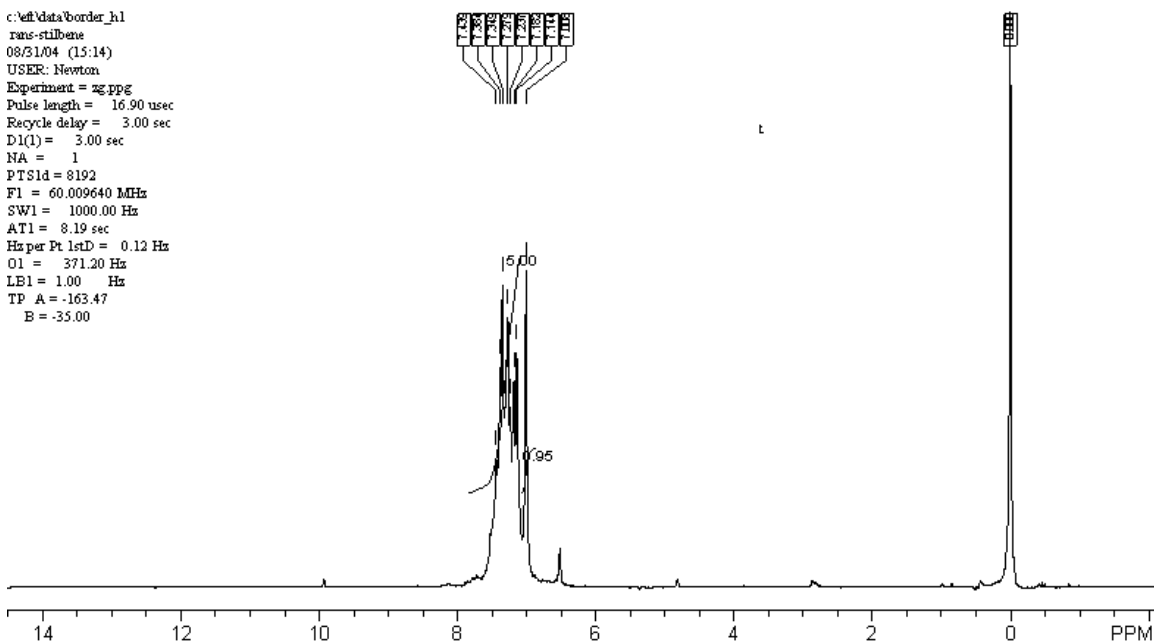


Figure 2. The ^1H NMR Spectrum of *trans*-Stilbene.

The gas chromatograph of my sample showed one major peak with a retention time of 6.04 minutes. The mass spectrum of this material had a molecular ion at $m/z = 180.1$ and a base peak at 179.3. A library search returned *trans*-stilbene as the closest match. The data are reproduced in Figure 3. There was no evidence for *cis*-stilbene in the gas chromatograph.

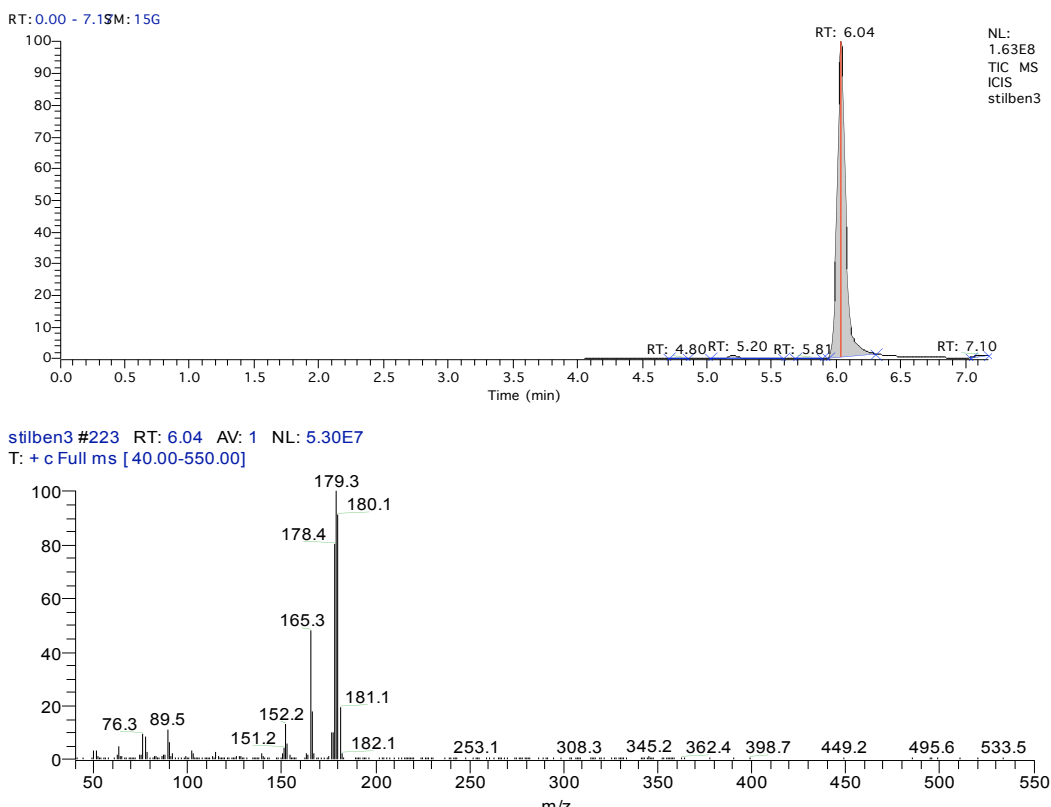


Figure 3. Top is the GC trace. Bottom is the mass spectrum of the peak with a retention time of 6.04 minutes.

