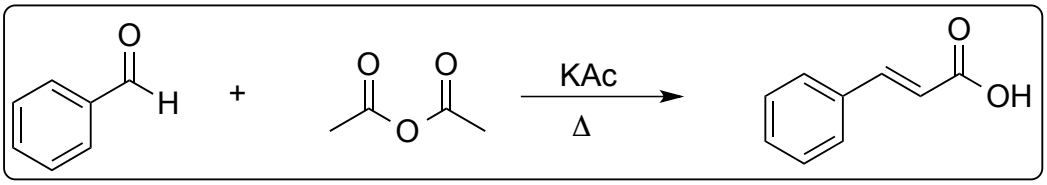
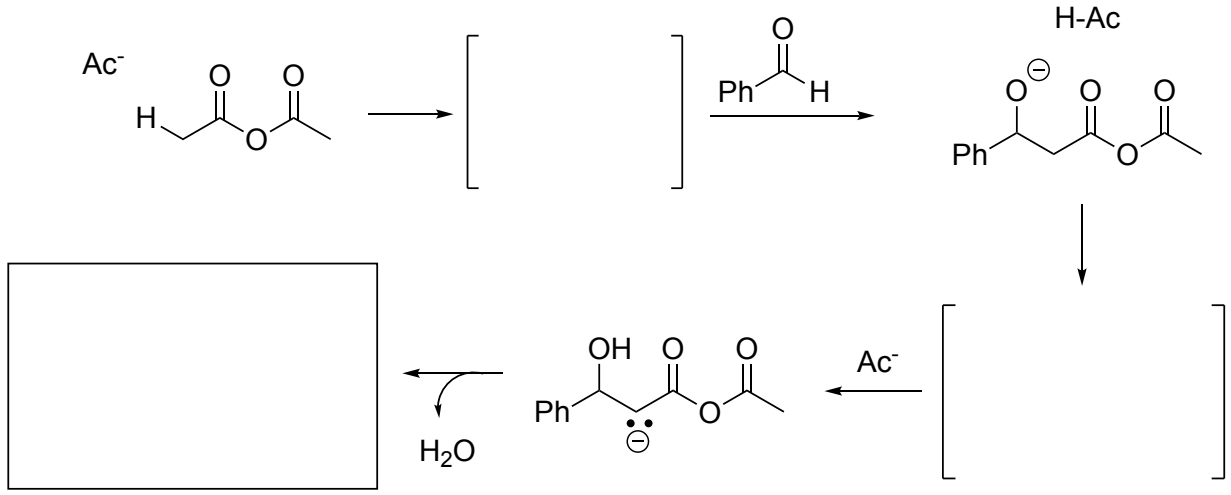
CHEM 110L, Experiment 1 – Perkin Condensation: Synthesis of *trans*-cinnamic acid

Perkin Condensation – [aromatic aldehyde + anhydride] – [acetate] = unsaturated carboxylic acid

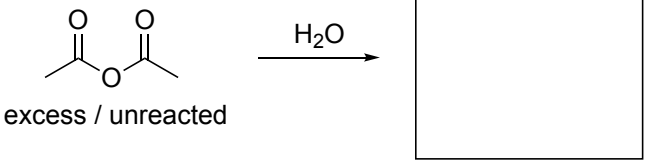


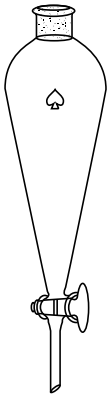
Reaction setup … Reflux for 1 hour



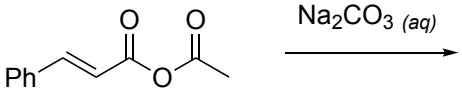
Reaction work-up … sep funnel action

1. Water



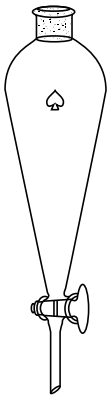


2. Aqueous sodium carbonate



Reaction work-up (cont'd)

3. *tert*-butyl methyl ether (BME) – organic solvent

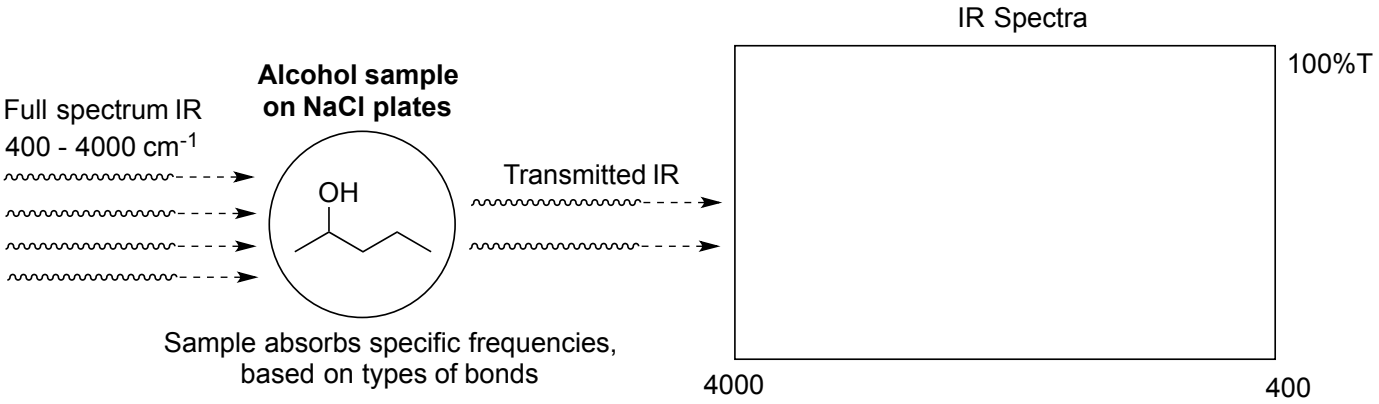


4. Concentrated hydrochloric acid (HCl) – precipitation & filtration

trans-cinnamate

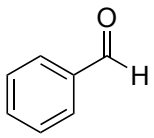
SPECTROSCOPY: CHARACTERIZATION OF ORGANIC COMPOUNDS

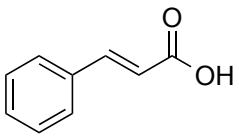
IR Spectroscopy



_____ Bond length corresponds to _____ stretching frequency (wavenumber cm^{-1}).

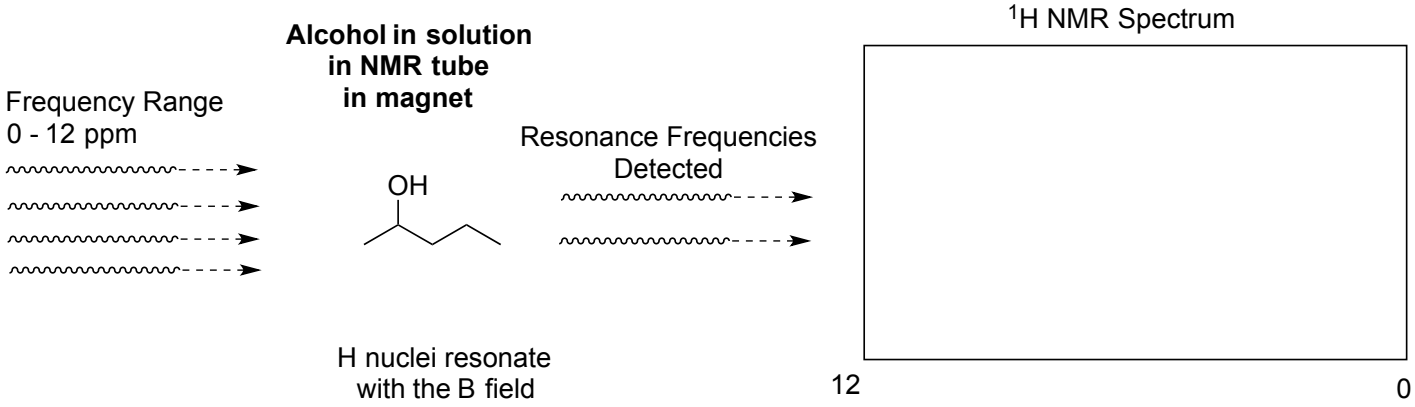
Where do you expect each compound to absorb in the IR spectrum?





How does conjugation affect stretching frequency?

Nuclear Magnetic Resonance (NMR) Spectroscopy



Shielding & Deshielding

- Electron-donating groups _____ the proton from the B field = _____ chemical shift

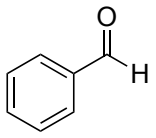
- Electron-withdrawing groups _____ the proton = _____ chemical shift

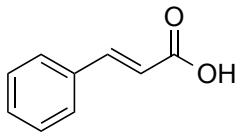
Predicting NMR Spectra from Structures

*Refer to spectroscopy tables online for values and/or nmrdb.org

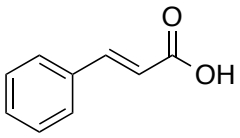
Proton ^{1}H NMR

- Chemical equivalence & Integration
- Chemical Shift
- Splitting

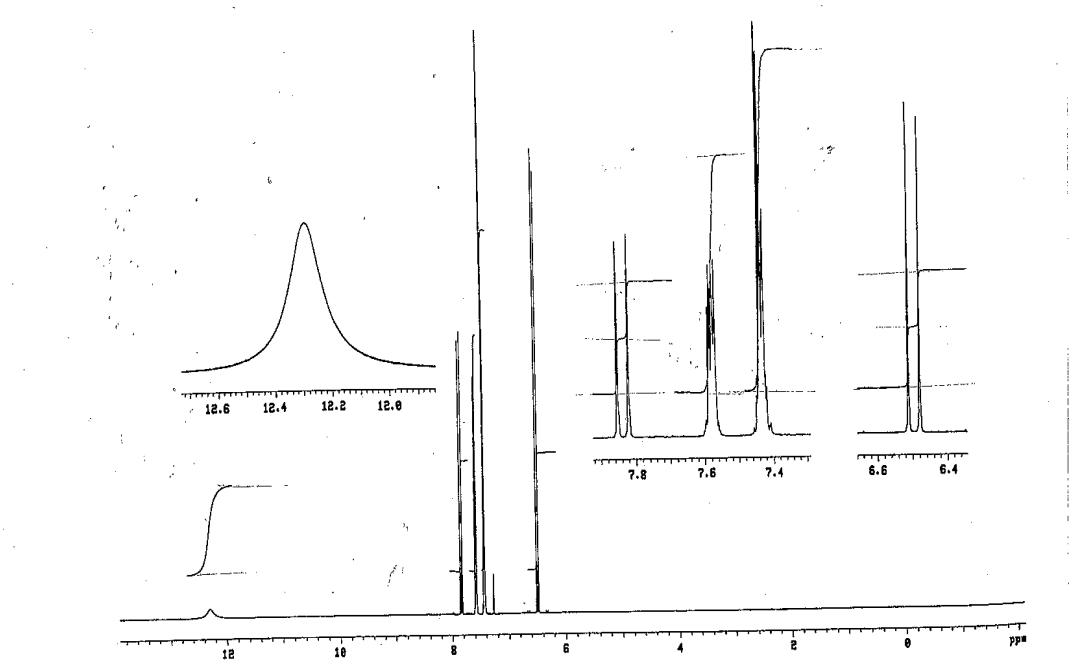




^{1}H NMR Analysis of *trans*-Cinnamic Acid



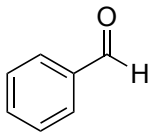
Signal	Integration (#H's)	Splitting	Chemical Shift, Expected (ppm)	Chemical Shift, Observed (ppm)
A				
B				
C				
D				
E				
F				

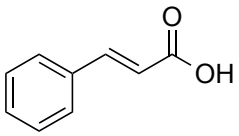


Literature spectra (Palleros text)

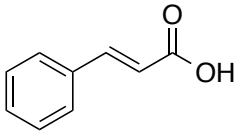
Carbon (^{13}C) NMR

- Chemical Shift
- Approximate Peak Height





^{13}C NMR Analysis of trans-cinnamic acid



Signal	Chemical Shift, Estimated	Chemical Shift, Observed
A'		
B'		
C'		
D'		
E'		
F'		
G'		

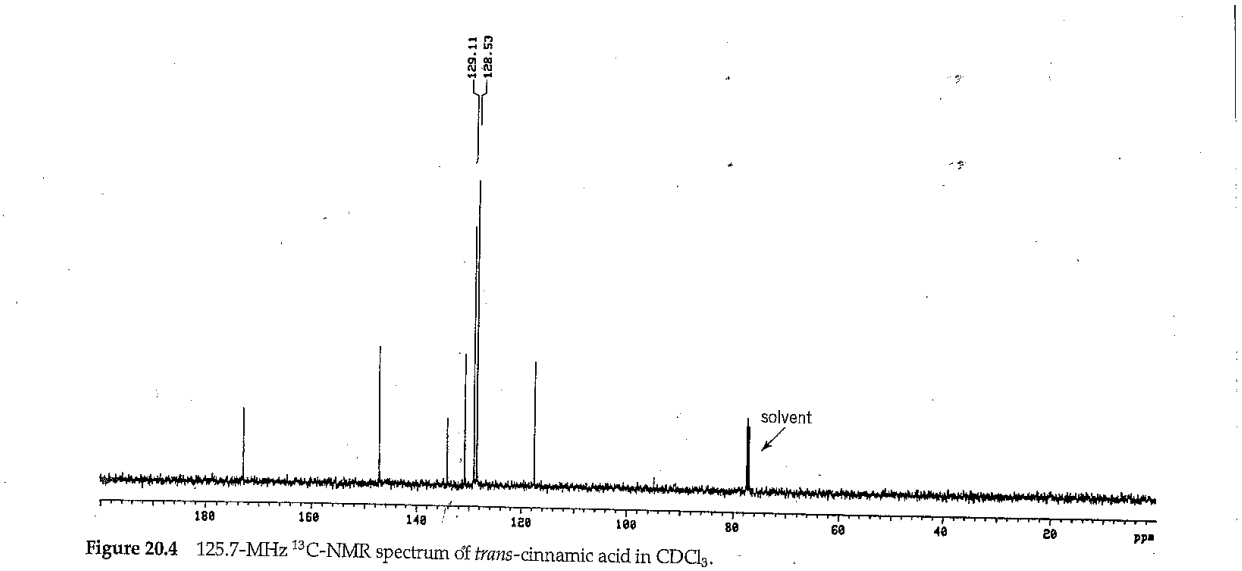


Figure 20.4 125.7-MHz ^{13}C-NMR spectrum of *trans*-cinnamic acid in $CDCl_3$.