# C8953
# NMR structural analysis - seminar
## Basic concepts & Vector model

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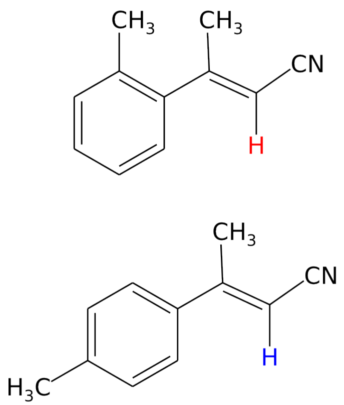
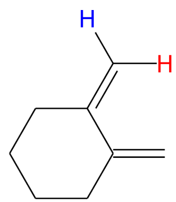
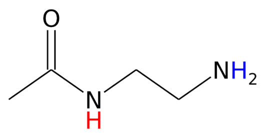
# Assign correct value of chemical shift to labelled NMR active atoms[1]:



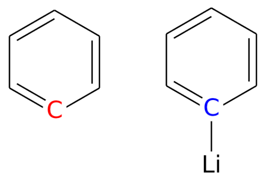
5.22 5.46



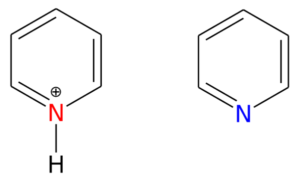
4.64 4.92



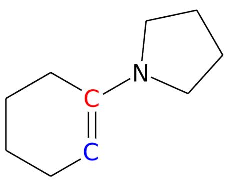
1.5 7.0



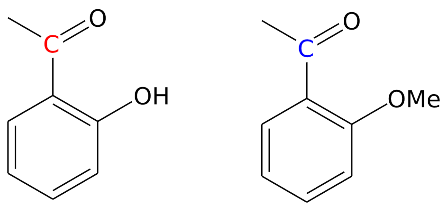
129 200



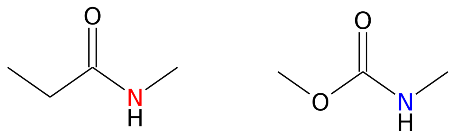
-166 -68



93 142

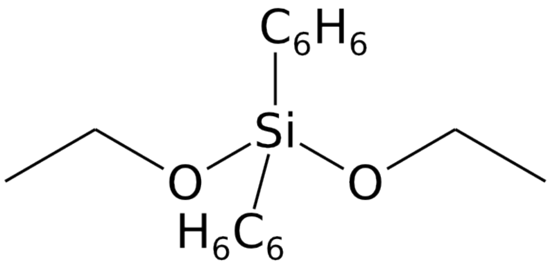


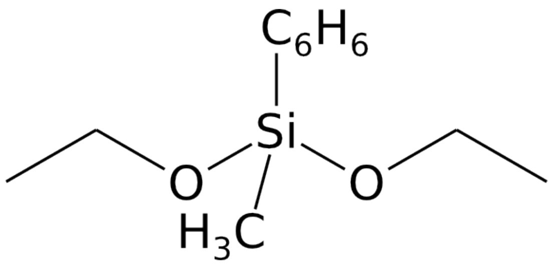
188 197

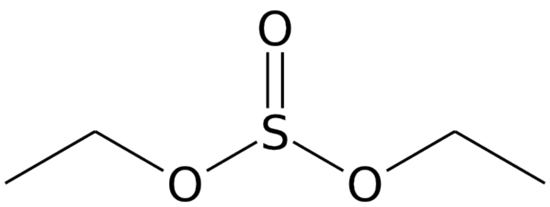


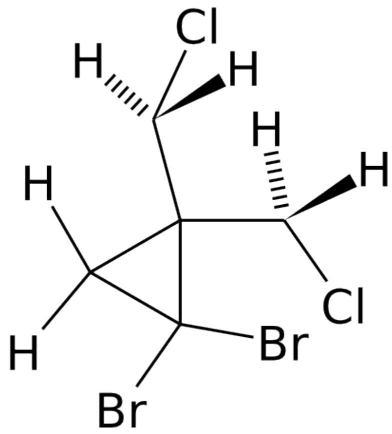
[1] http://www.chem.wisc.edu/areas/reich/chem605/

# Diastereotopicity[1]





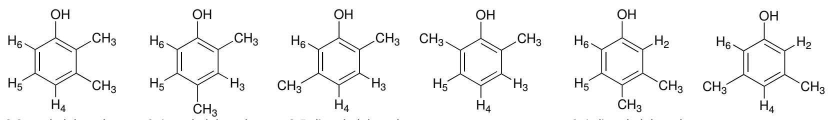


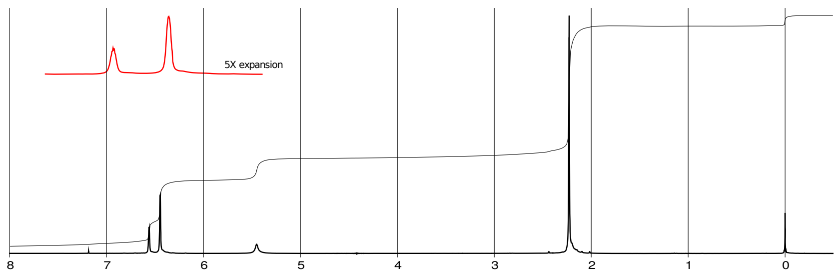


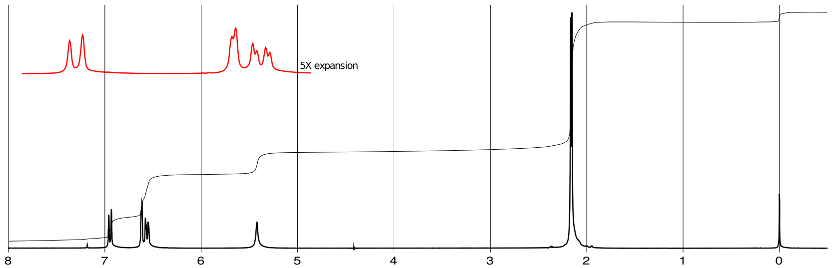
[1] http://www.chem.wisc.edu/areas/reich/chem605/

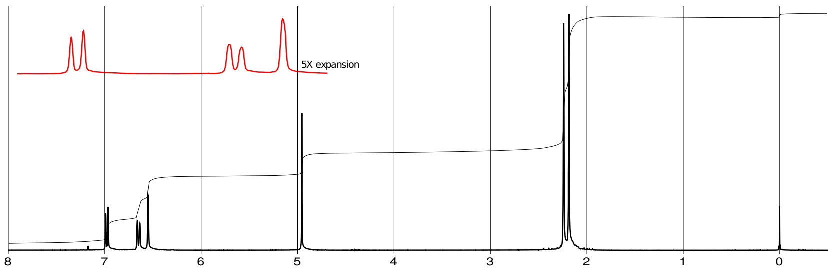
# Determination of regioisomers

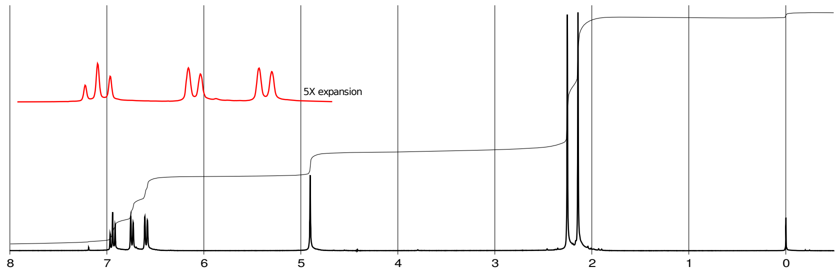
300 MHz ¹H NMR spectra in $CDCl_3$

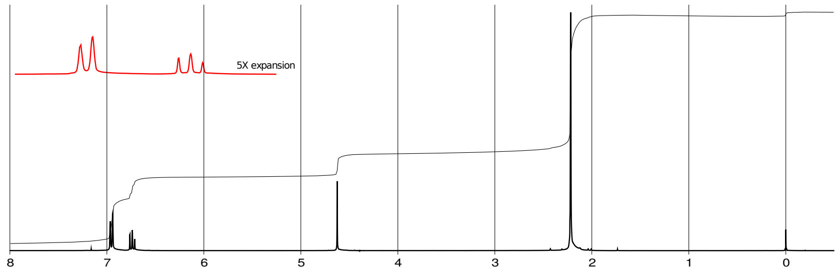


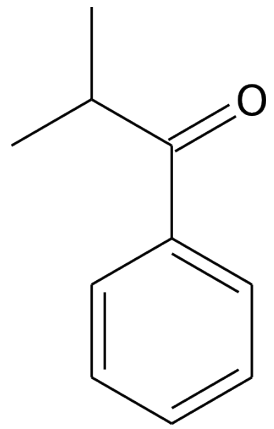



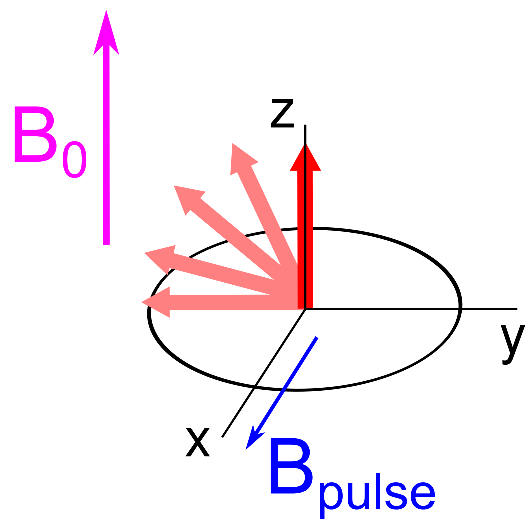



Draw the estimate of $^{13}C$ NMR spectrum (with and without $^{1}H$ decoupling) and APT experiment:



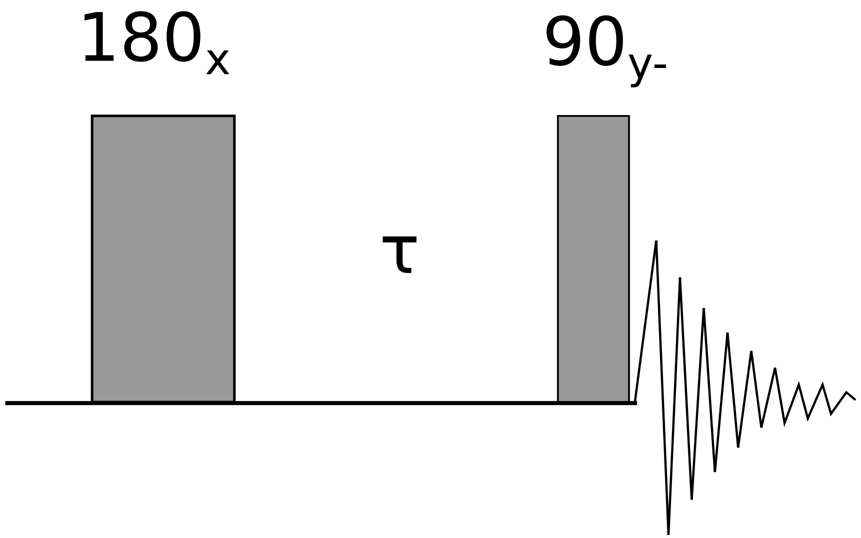
# Analysis of simple pulse sequences using vector model

- simple model based on rotation of the vector of bulk magnetization in the plane perpendicular to the vector of magnetic field, direction is determined by the "right-hand rule"
- NMR signal is detectable only as coherent magnetization oscillating in *xy* plane
- the free precession $\omega$ (due to the $B_0$) of magnetization vector is eliminated by introducing rotating frame $\omega_0 \Rightarrow$ magnetic field of excitation pulses ($B_1$) is motionless and the individual resonance frequencies differs in so called offset $\Omega_i = \omega_i - \omega_0$
- applicability of vector model is rather limited to simple single-quantum experiments without transfer of polarisation



# $T_1$ relaxation

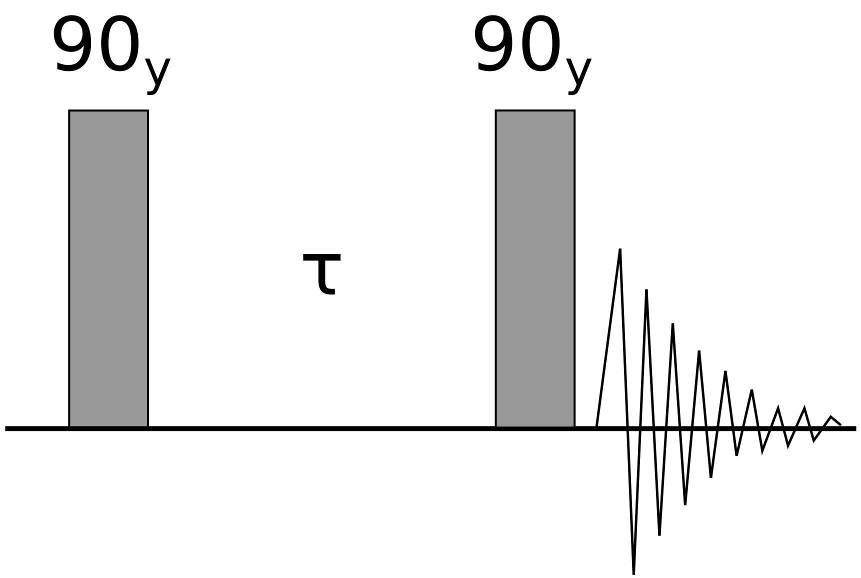
Apply following sequence (inversion recovery) to isolated spin characterized by **a)** $\tau = 2 * T_1$ and **b)** $\tau = 0.2 * T_1$. Draw semi-quantitatively resulting spectrum.



# 1-$\overline{1}$ sequence

Draw the evolution of macroscopic magnetization through the sequence:
**90(y) - $\tau$ - 90(y) - aq**
Consider the evolution of an isolated spin due to the chemical shift.

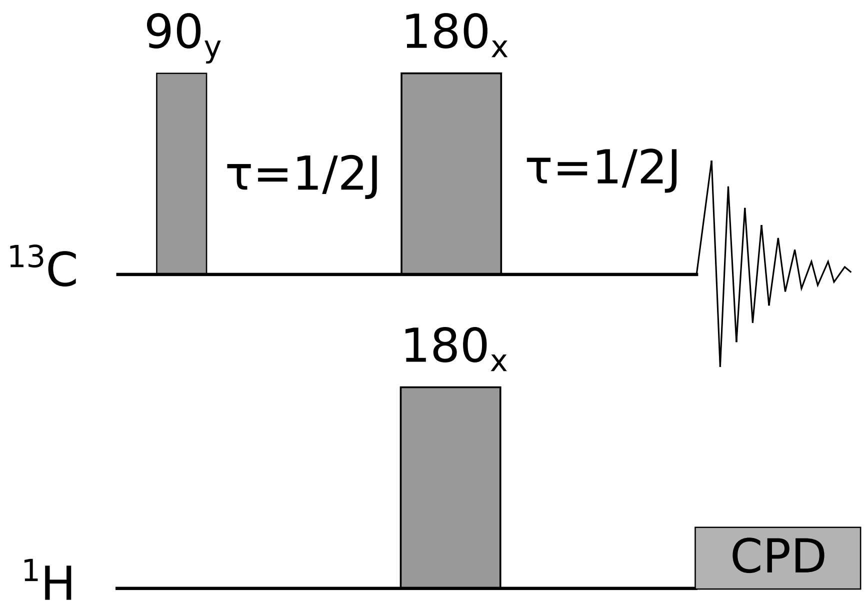
1. How does the result differ for the following offsets: $\Omega\tau = 0, \pi/2, \pi$.
2. Draw lineshapes of resulting signal assuming the a) $y+$ b) $x+$ corresponds to zero phase of receiver.



# Heteronuclear spin echo

By using vector diagrams determine the result of attached pulse sequence.

1. **Ignore 180 pulse** in hydrogen channel for isolated spin systems **a)** $^{13}C$-$^{1}H$ and **b)** $^{13}C$-$^{1}H_2$. Explain the role of CPD block.
2. Lets consider **the complete sequence** and isolated spin systems **a)** $^{13}C$-$^{1}H$ and **b)** $^{13}C$-$^{1}H_2$.



# Next topic

2D NMR - homonuclear experiments