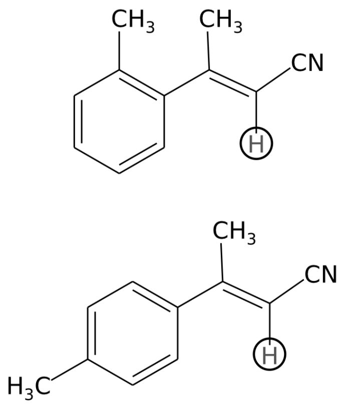
C8953
NMR structural analysis - seminar
Few Basic Concepts & Vector model

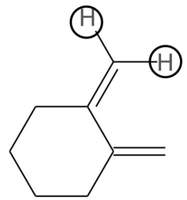
Kateřina Peterková, Aleš Novotný
423977@mail.muni.cz

March 13, 2019

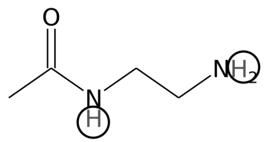
Assign correct value of chemical shift to labelled NMR active atoms[1]:



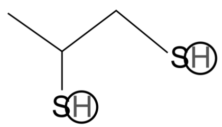
5.22 5.46



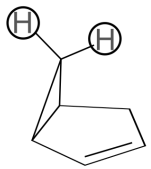
4.64 4.92



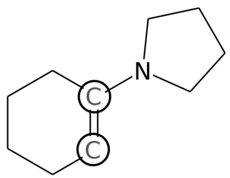
1.5 7.0



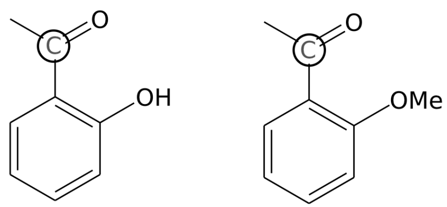
1.65(t) 1.85(d)



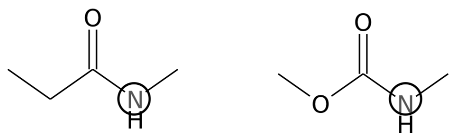
-0.17 0.73



93 142

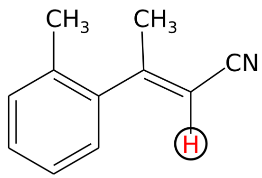


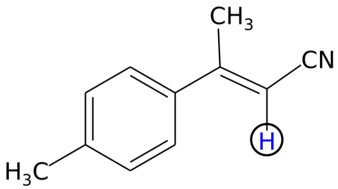
188 197



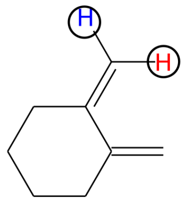
[1] http://www.chem.wisc.edu/areas/reich/chem605/

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

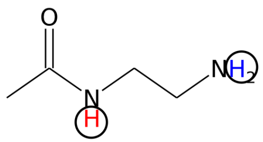




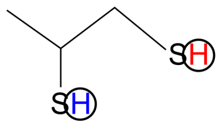
5.22 5.46



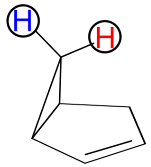
4.64 4.92



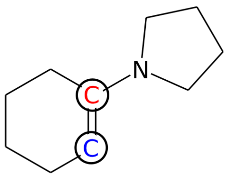
1.5 7.0



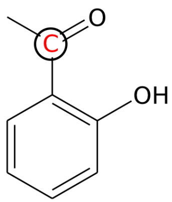
1.65(t) 1.85(d)

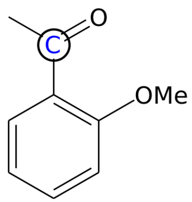


-0.17 0.73

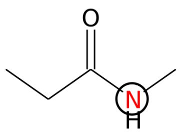


93 142

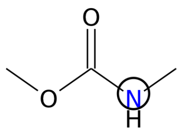




188 197

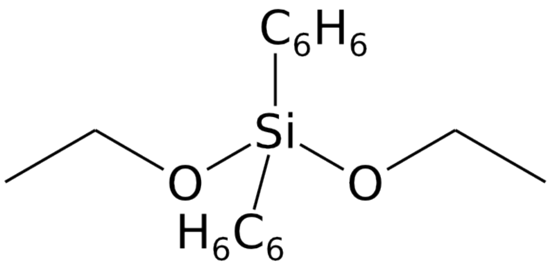


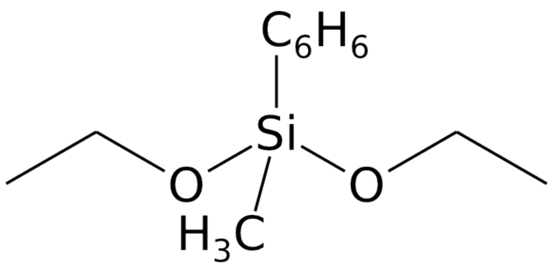
>

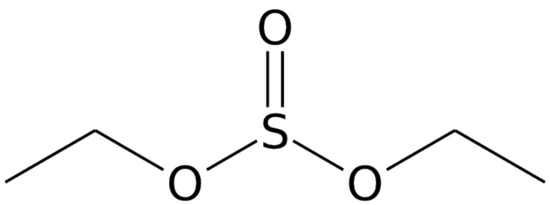


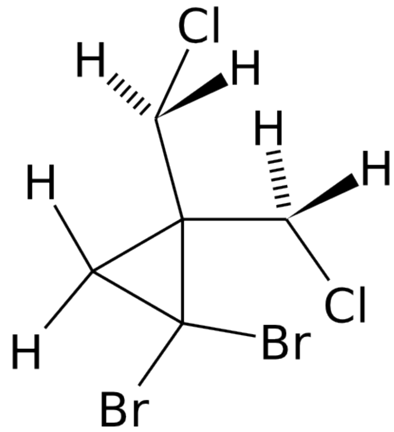
[1] http://www.chem.wisc.edu/areas/reich/chem605/

Diastereotopicity[1] Determine the equivalency of geminal protons



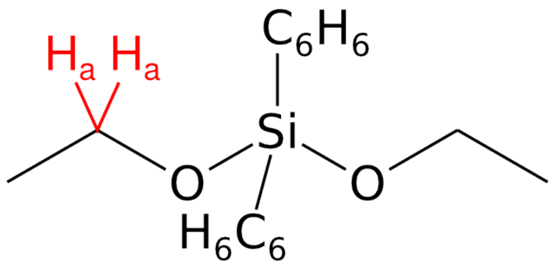


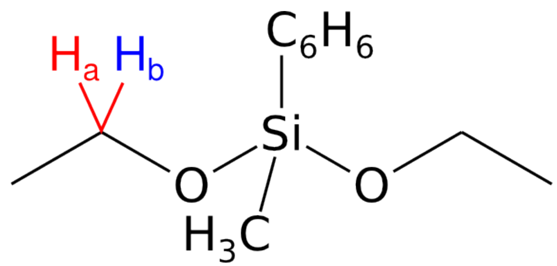


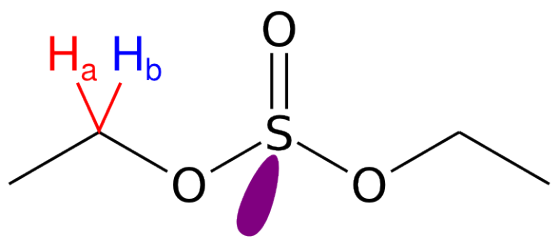


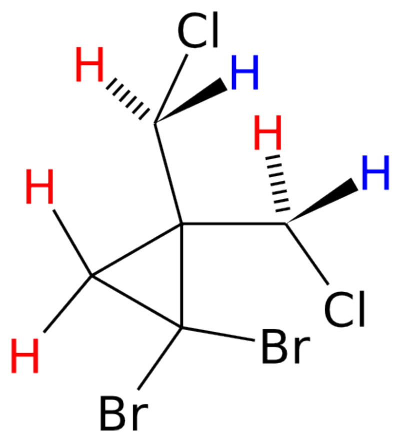
[1] http://www.chem.wisc.edu/areas/reich/chem605/

Diastereotopicity[1] Determine the equivalency of geminal protons



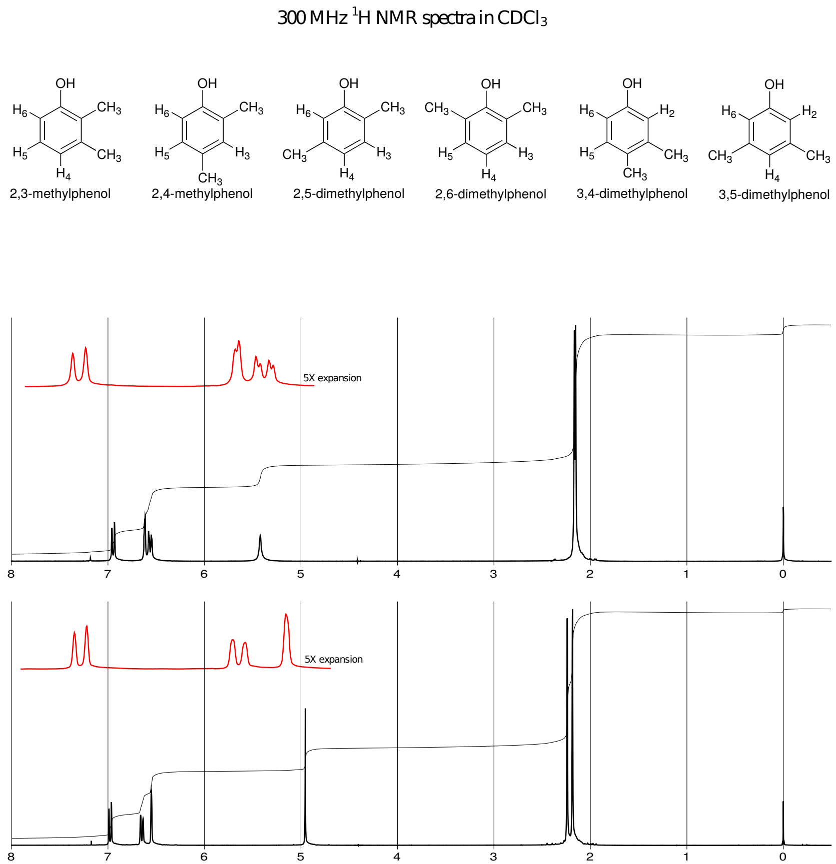


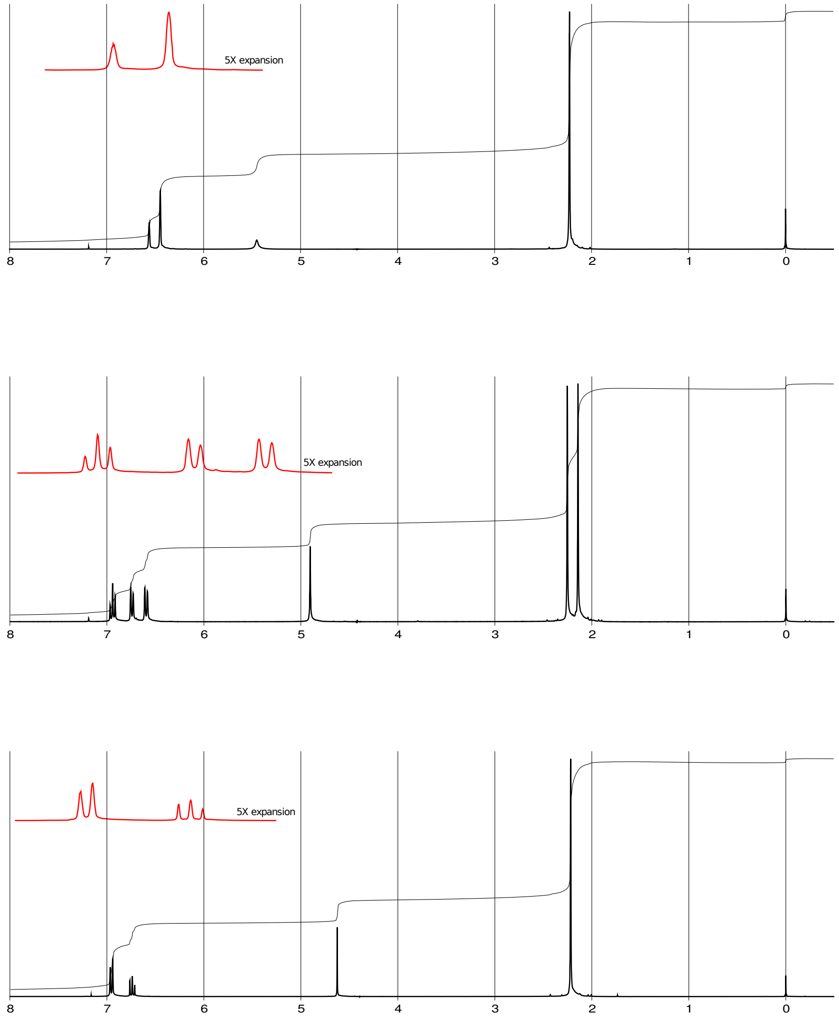




[1] http://www.chem.wisc.edu/areas/reich/chem605/

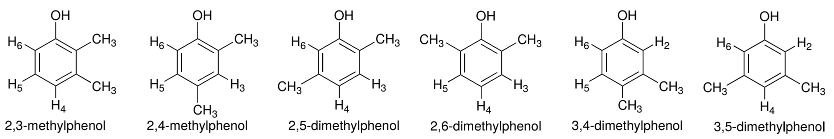
Determination of regioisomers - note number of signals, splitting and δ

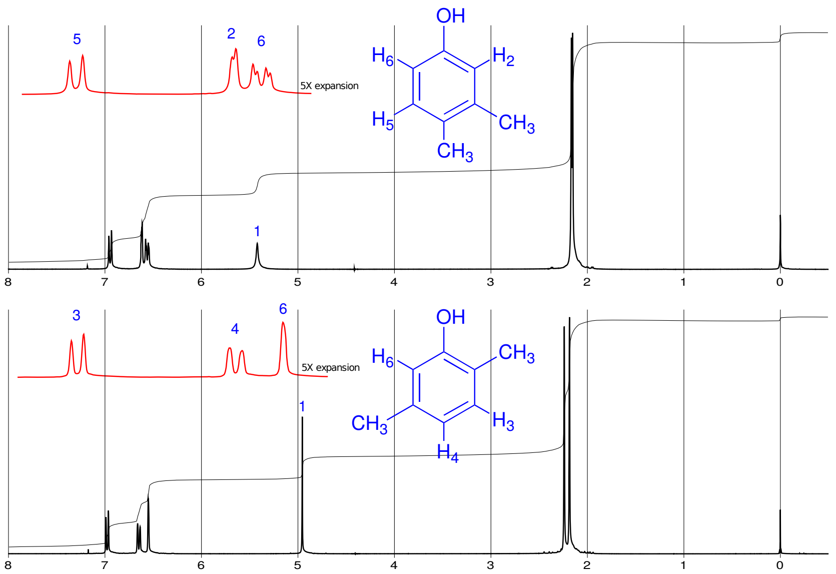


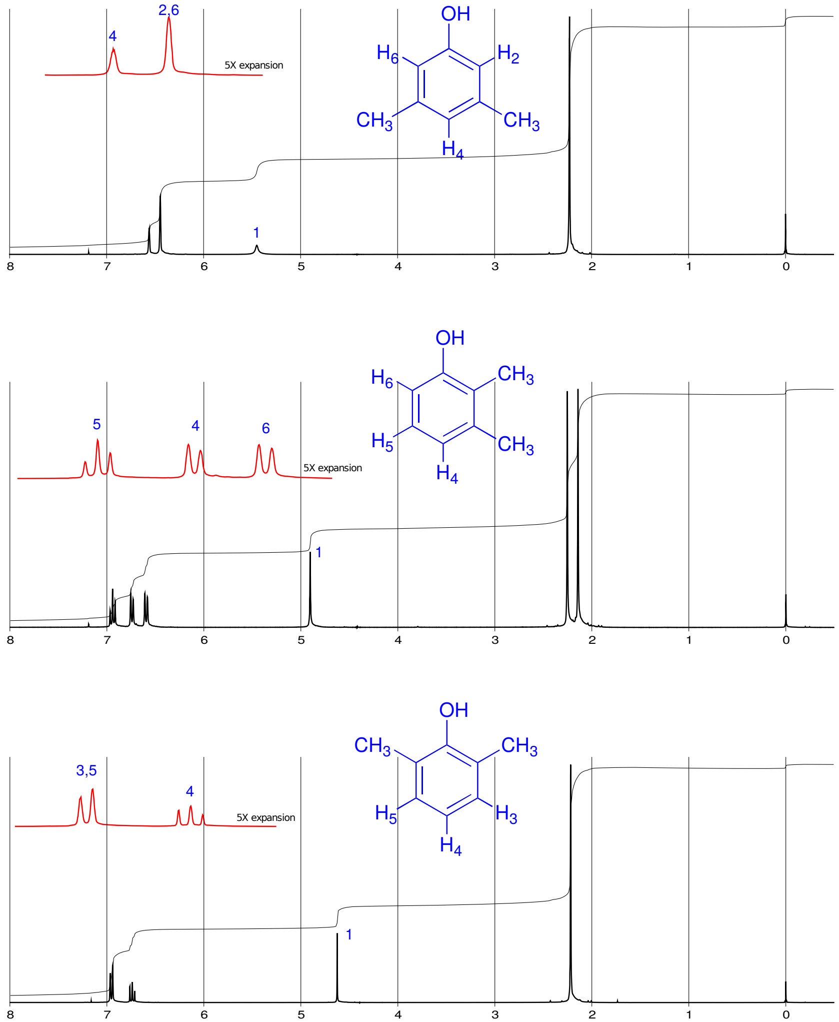


Determination of regioisomers - note number of signals, splitting and δ

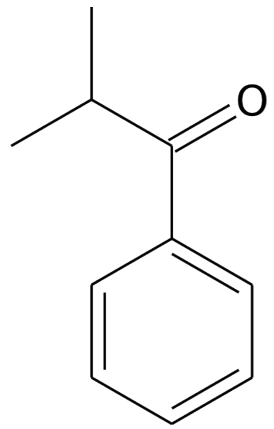
300 MHz ^{1}H NMR spectra in $CDCl_3$



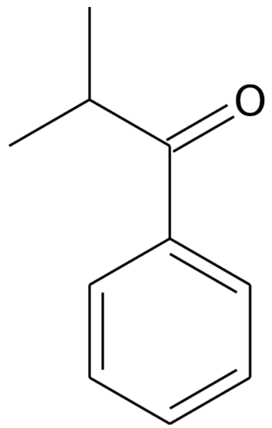


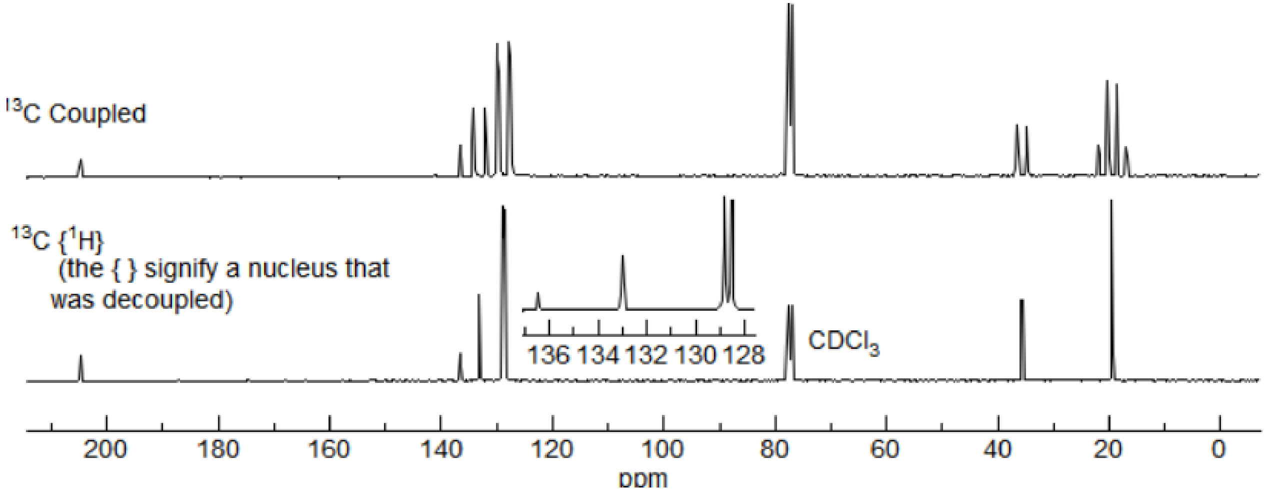


Draw the estimate of ^{13}C NMR spectrum (with and without ^{1}H decoupling)



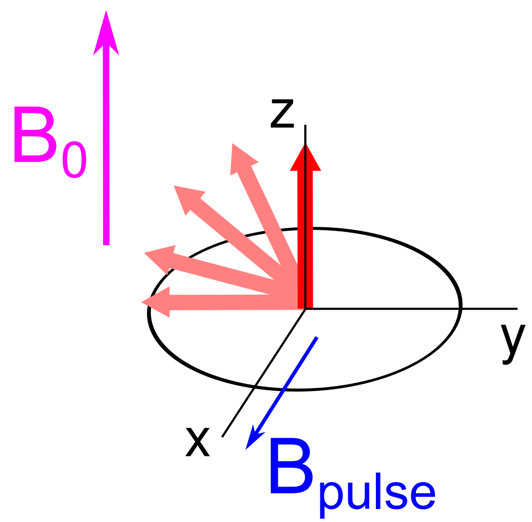
Draw the estimate of ^{13}C NMR spectrum (with and without ^{1}H decoupling)





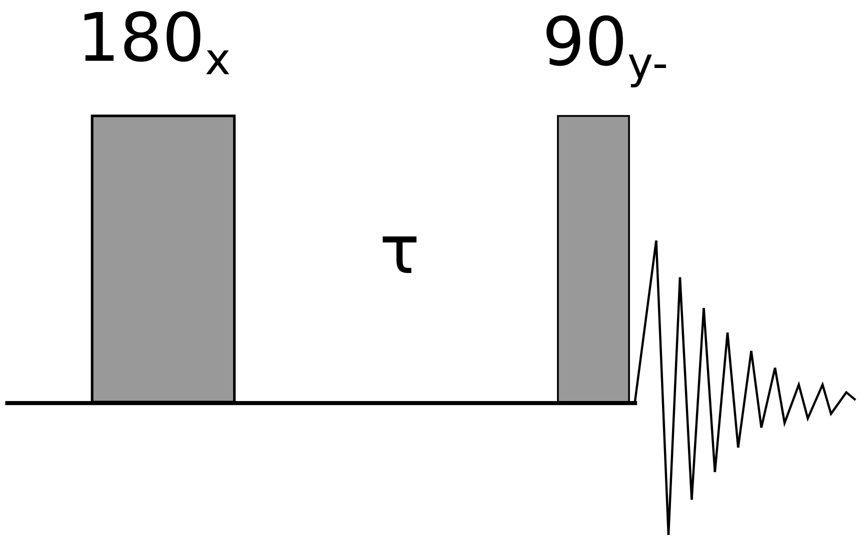
Analysis of simple pulse sequences using vector model

- 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 ω (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



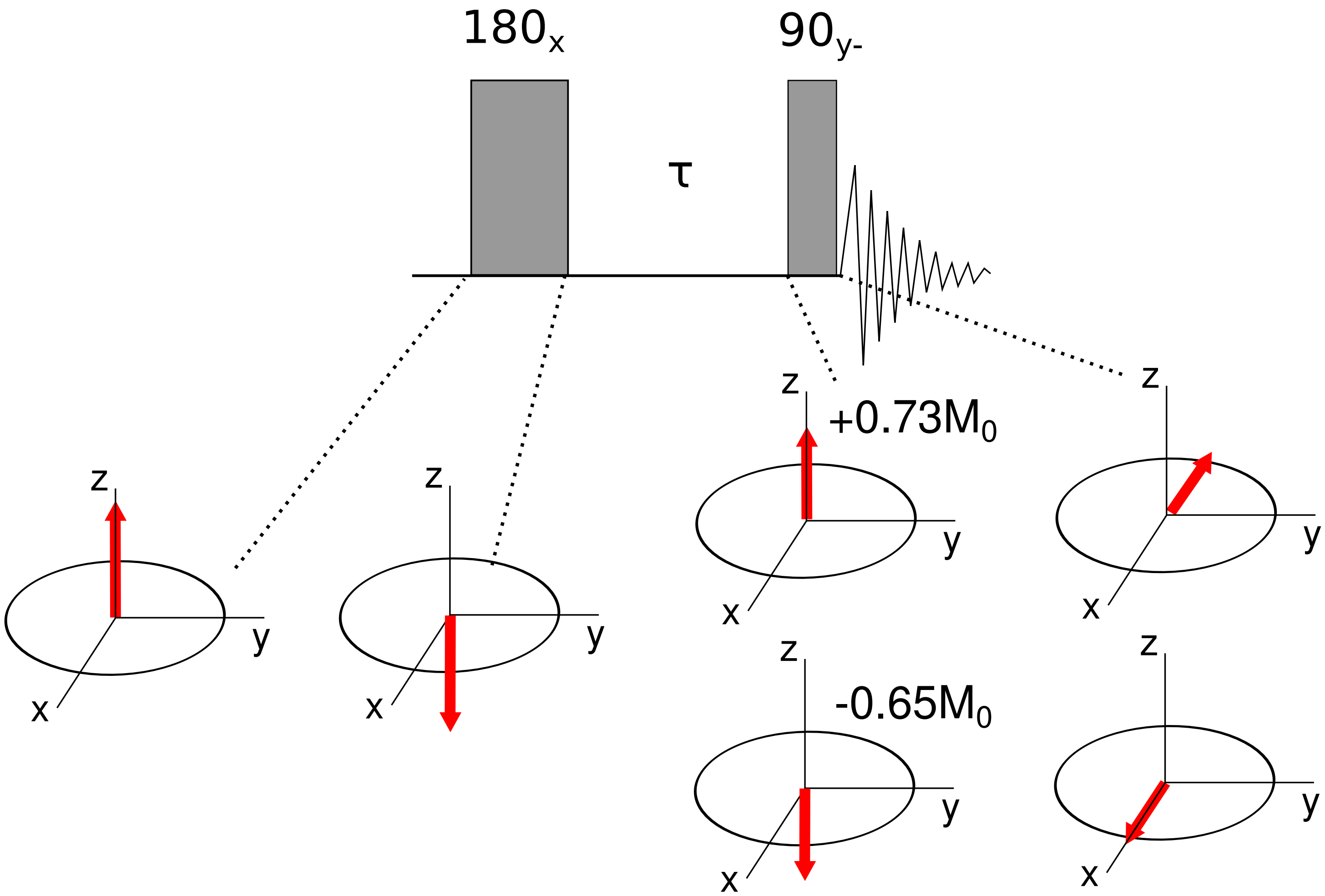
T_1 relaxation

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.



T_1 relaxation

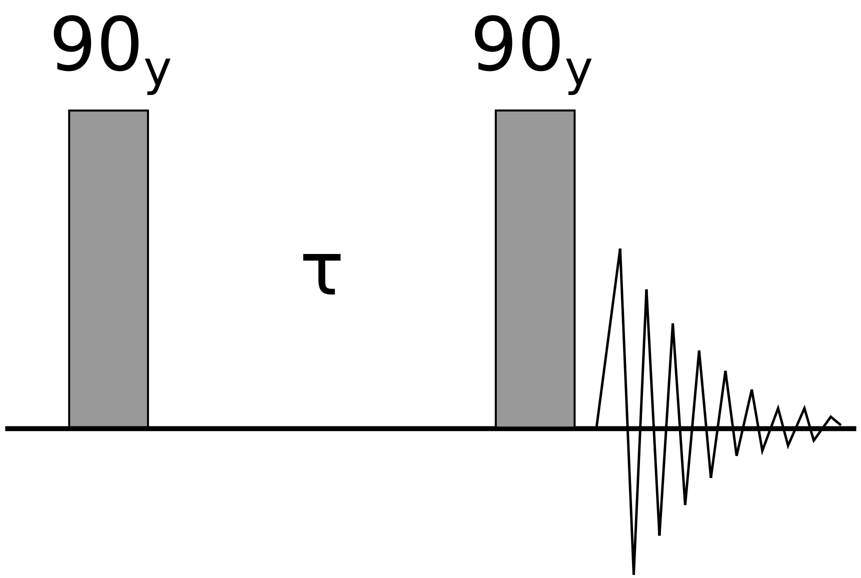
Apply following sequence (inversion recovery) to isolated spin characterized by **a)** $\tau = 2 * T_1$ and **b)** $\tau = 0.2 * T_1$. Draw semi-quantitatively resulting spectrum.



1-$\bar{1}$ sequence

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

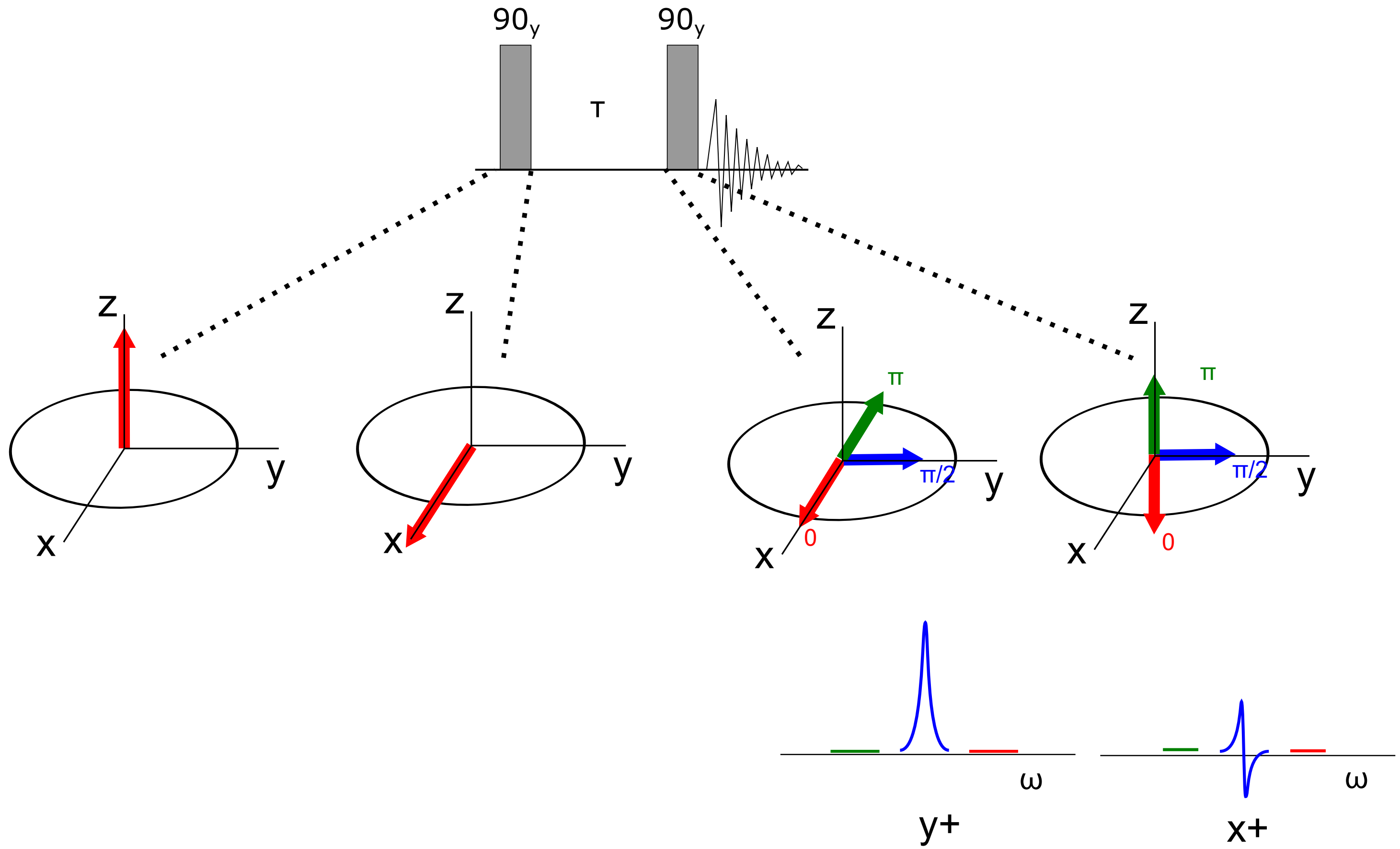
1. How does the result differ for the following offsets: $\Omega\tau = 0, \pi/2, \pi$.
2. Draw lineshapes of resulting signal assuming the a) $y+$ b) $x+$ corresponds to zero phase of receiver (prior phase correction).



$1\text{-}\bar{1}$ sequence

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

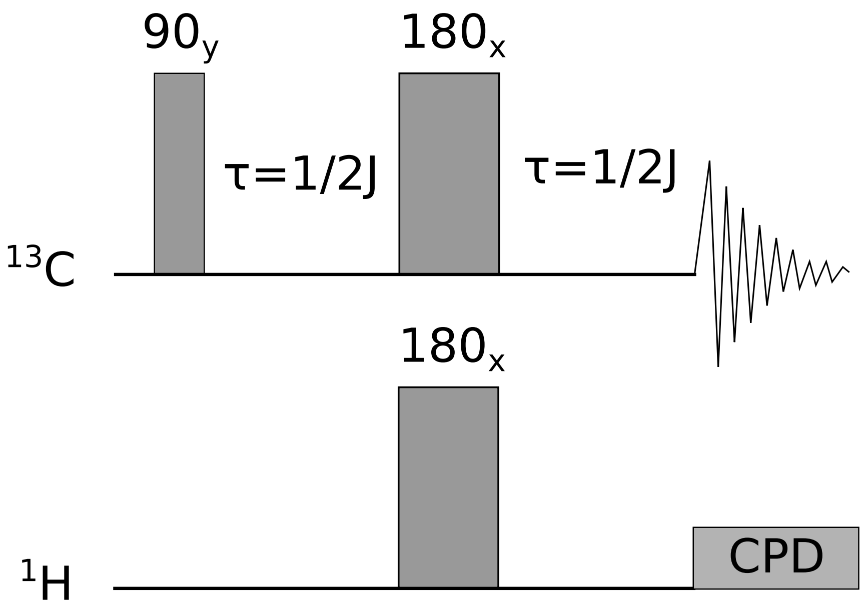
1. How does the result differ for the following offsets: $\Omega\tau = 0, \pi/2, \pi$.
2. Draw lineshapes of resulting signal assuming the a) $y+$ b) $x+$ corresponds to zero phase of receiver (prior phase correction).



Heteronuclear spin echo

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

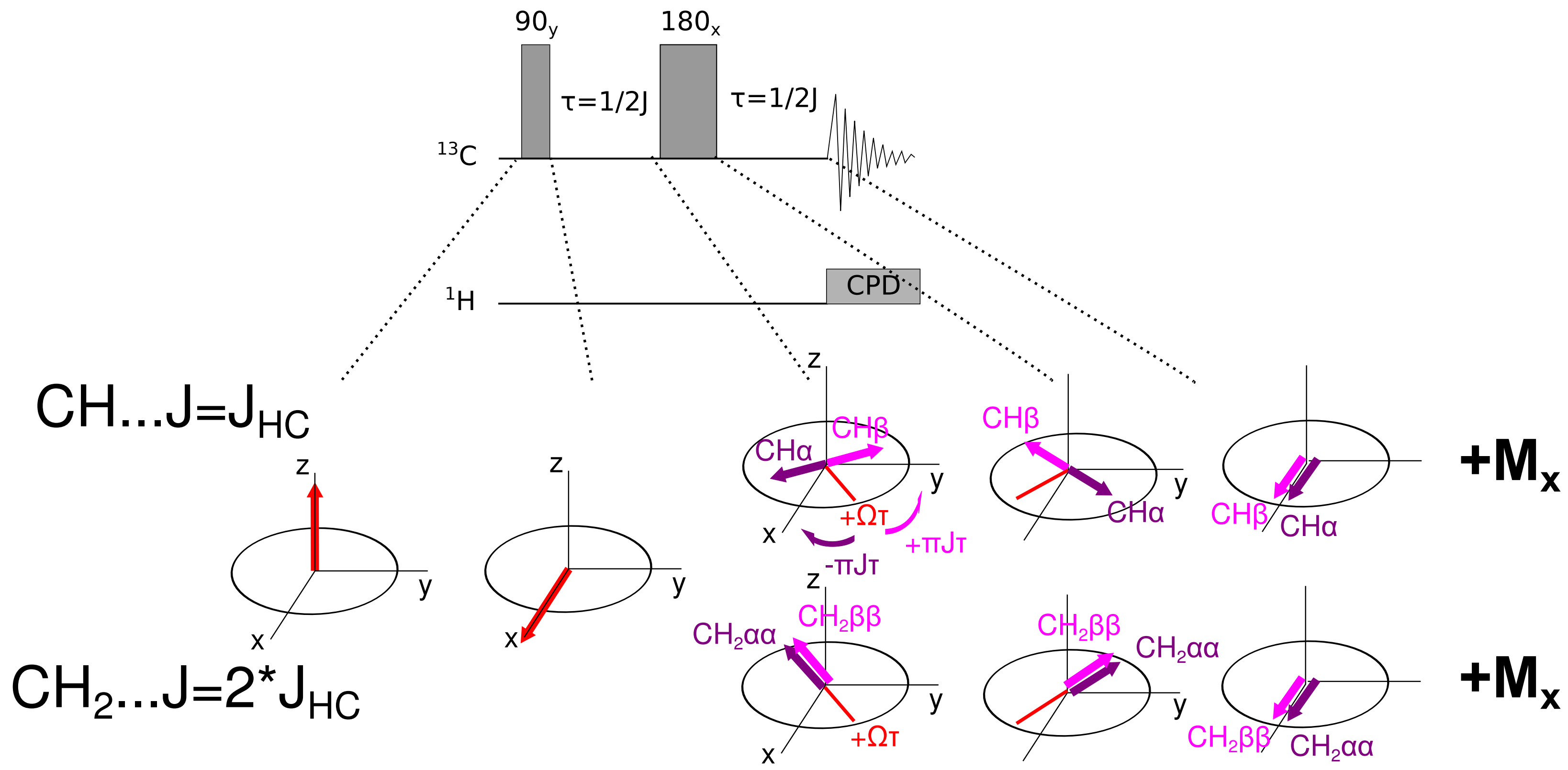
1. **Ignore 180 pulse** in hydrogen channel for isolated spin systems **a)** ^{13}C-^{1}H and **b)** ^{13}C-$^{1}H_2$. Explain the role of CPD block.
2. Lets consider **the complete sequence** and isolated spin systems **a)** ^{13}C-^{1}H and **b)** ^{13}C-$^{1}H_2$.



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.



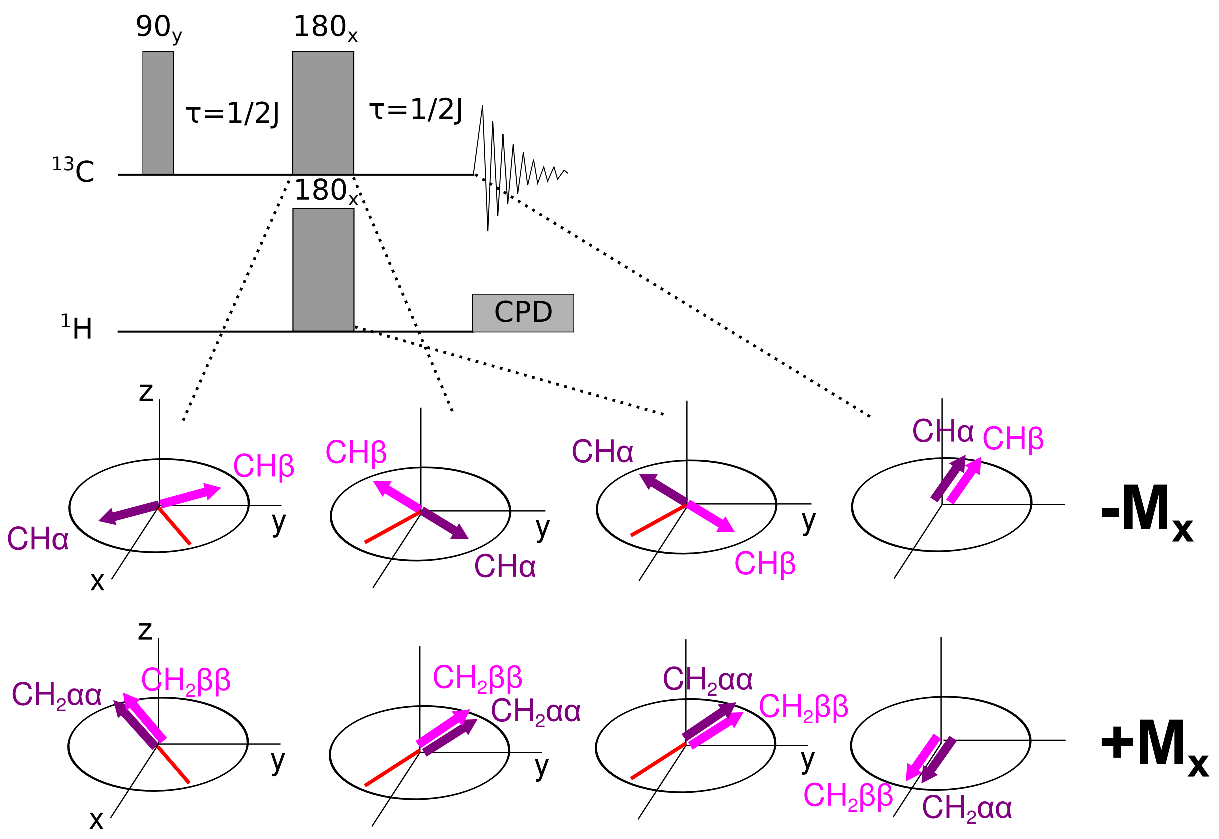
Heteronuclear spin echo

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

2. Lets consider **the complete sequence** and isolated spin systems **a)** ^{13}C-^{1}H and **b)** ^{13}C-$^{1}H_2$.

CH...J=J_{HC}

CH$_2$...J=2*J_{HC}



Next topic

edited 1D 13C spectra, 2D NMR - homonuclear experiments