CHM 4380 / 8309C Assignment #1 of 2

Answer all questions neatly and independently, showing all your work. Total = 60 points

Assigned: **Tuesday November 15**

Due: **Tuesday November 22 (at the beginning of class)**

1. ^{11}B is an NMR active nucleus.

(a) How many Zeeman energy levels does ^{11}B have? (2 points)

(b) How many lines would you expect to see in the ^{11}B NMR spectrum of dissolved BCl_3? (2 points)

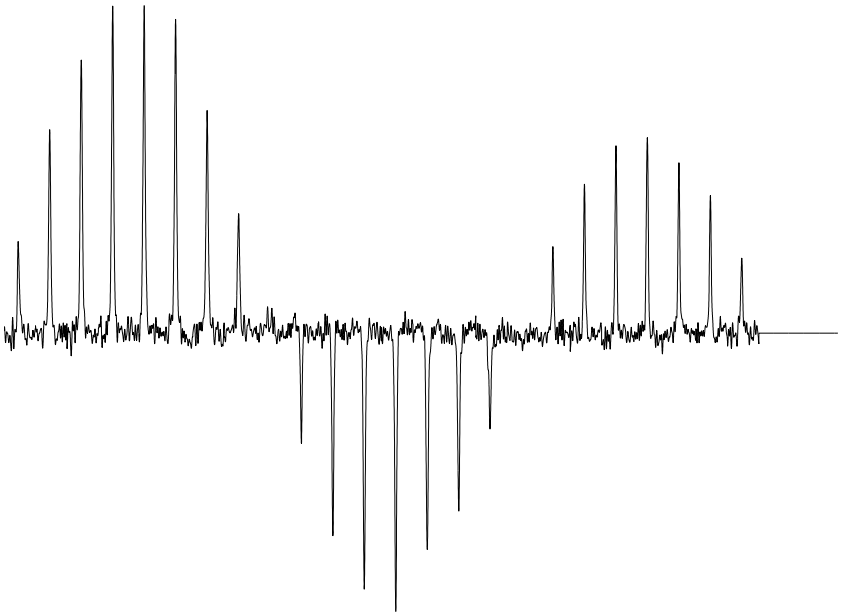
(c) In a particular NMR spectrometer, the Larmor frequency for ^{11}B is 192.546 MHz. What is the strength of the magnet in Tesla? (2 points)

(d) What is the ^{1}H NMR frequency for the NMR instrument described in (c)? (2 points)

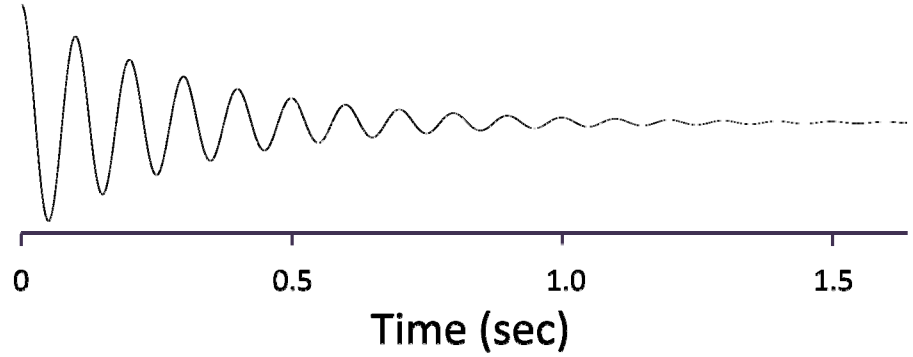
2. A Varian INOVA 400 spectrometer was used to measure the ^{13}C NMR spectrum of hexamethyl benzene. The spectrum consisted of two lines separated by 11,559.26 Hz. The chemical shift of the methyl carbon was 17.3 ppm. What is the chemical shift of the aromatic carbons? (4 points)

3. Two ^{1}H NMR spectra of $CHCl_3$ were collected. All acquisition parameters were the same for both spectra except that In the first spectrum, the ^{1}H NMR signal was on-resonance and in the second spectrum it was 50 kHz off resonance. The intensity of the $CHCl_3$ signal was 30% lower in the second spectrum compared to the first. Explain. (4 points)

4. A series of single scan ^{2}H NMR spectra for D_2O were collected with pulse durations ranging from 0.5 μsec to 12.0 μsec in 0.5 μsec steps. The spectra, allowed for complete relaxation, were plotted side-by-side and are displayed below. What is the duration of the 270°, 360°, 180° and 90° pulses (to the nearest 0.5 μsec)? (4 points)



5. The ^{7}Li FID of 1M lithium chloride was measured on a Bruker AVANCE 600 MHz NMR spectrometer and is shown below.



(a) What is the difference (in ppm) between the ^{7}Li NMR signal of 1M LiCl and the carrier frequency? (4 points)

(b) Draw a sketch of the FID if the carrier frequency was coincident with the ^{7}Li signal. (2 points)

6. Draw vector diagrams for the following pulse train applied to a single resonance at equilibrium.

$$(\pi/2)_{x} - (\pi/2)_{-x} - (\pi)_{y} - (\pi/2)_{y} - (\pi/2)_{-x} - (\pi)_{x} - (\pi/2)_{x} - (\pi/2)_{-y} - (\pi)_{y}$$

On which axis does the magnetization reside after the pulse train? (4 points)

7. The following pulse sequence was applied to a water sample in a magnetic field at the ^{1}H Larmor frequency.

$$(\pi)_x - \tau - \left(\pi/2\right)_x - \ acquire\ signal\ on\ (-y)\ axis$$

Many spectra were acquired with the delay, τ, being varied from 0 to tens of seconds.

(a) Draw vector diagrams for this sequence for a very short and a very long value of τ. (6 points)

(b) Draw a sketch of the NMR spectrum with τ = 0 and τ = ∞. (4 points)

(c) Make a general sketch of the intensity of the NMR line vs. τ. (4 points)

(d) What parameter do you think could be measured by using a pulse sequence like this? (2 points)

8. Electronic components such as capacitors, transistors and other integrated circuits used to make NMR spectrometers have different efficiencies at different Larmor frequencies. Explain in a few sentences what NMR instrument manufacturers do to avoid the NMR spectrometers being less efficient for some nuclei and more efficient for others. (4 points)

9. Your department installed an NMR spectrometer in 1991. During the installation, the service engineer measured the absolute ^{1}H frequency of HDO to be 500.1399741 MHz. Twenty years later, in 2011, you measure the absolute frequency of HDO on the same spectrometer to be 500.1149352 MHz.

(a) The downward drift of the magnet is responsible for the change in the absolute frequency of HDO. What is the difference in field strength (in Tesla) for the magnet in 2011 compared to the day it was installed in 1991? (2 points)

(b) What would one have to do to make the absolute frequency of HDO the same as it was in 1991? (2 points)

(c) Assuming a linear field drift, how long would it take the magnet to drift down to zero? (2 points)

10. A student collected a simple ^{13}C NMR spectrum for 4.5 hours with 4096 scans. The acquisition time for a single scan was 1 second. The pulse duration was 6 μsec. How much time was permitted for relaxation in each scan? (4 points)