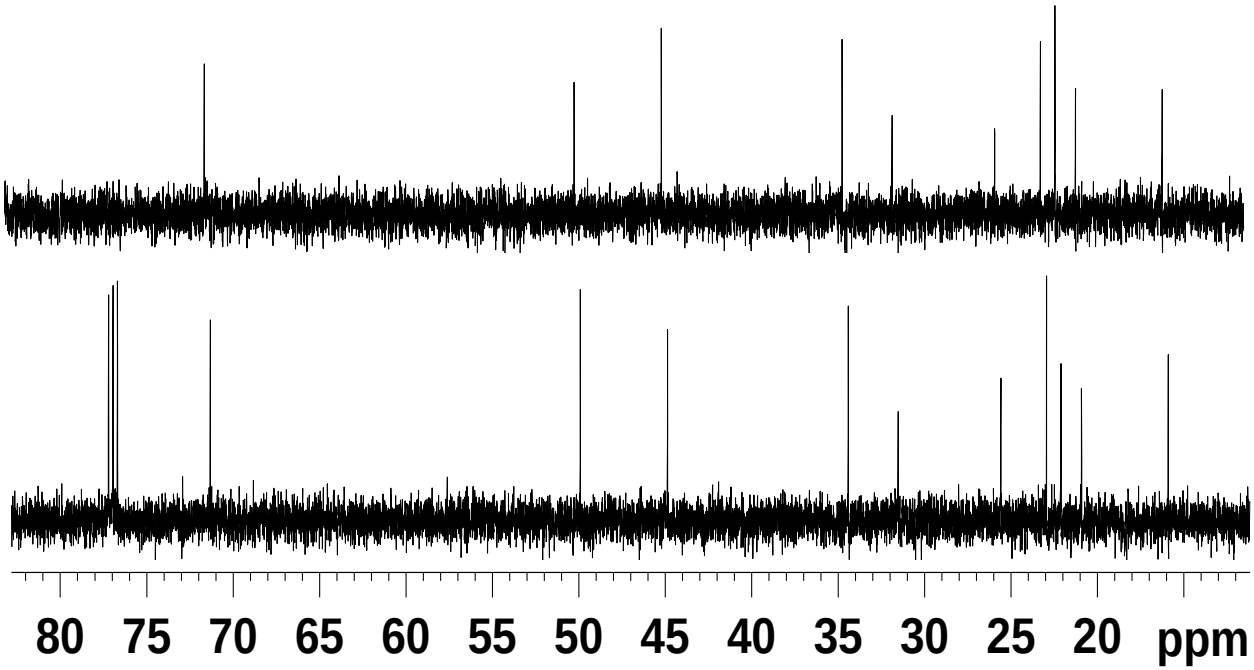
CHM 4380 / 8309C Assignment #2 of 2

Answer all questions neatly and independently. Show all work. Total = 76 points
Assigned: **Friday November 25**
Due: **Friday December 2 (at the beginning of class**

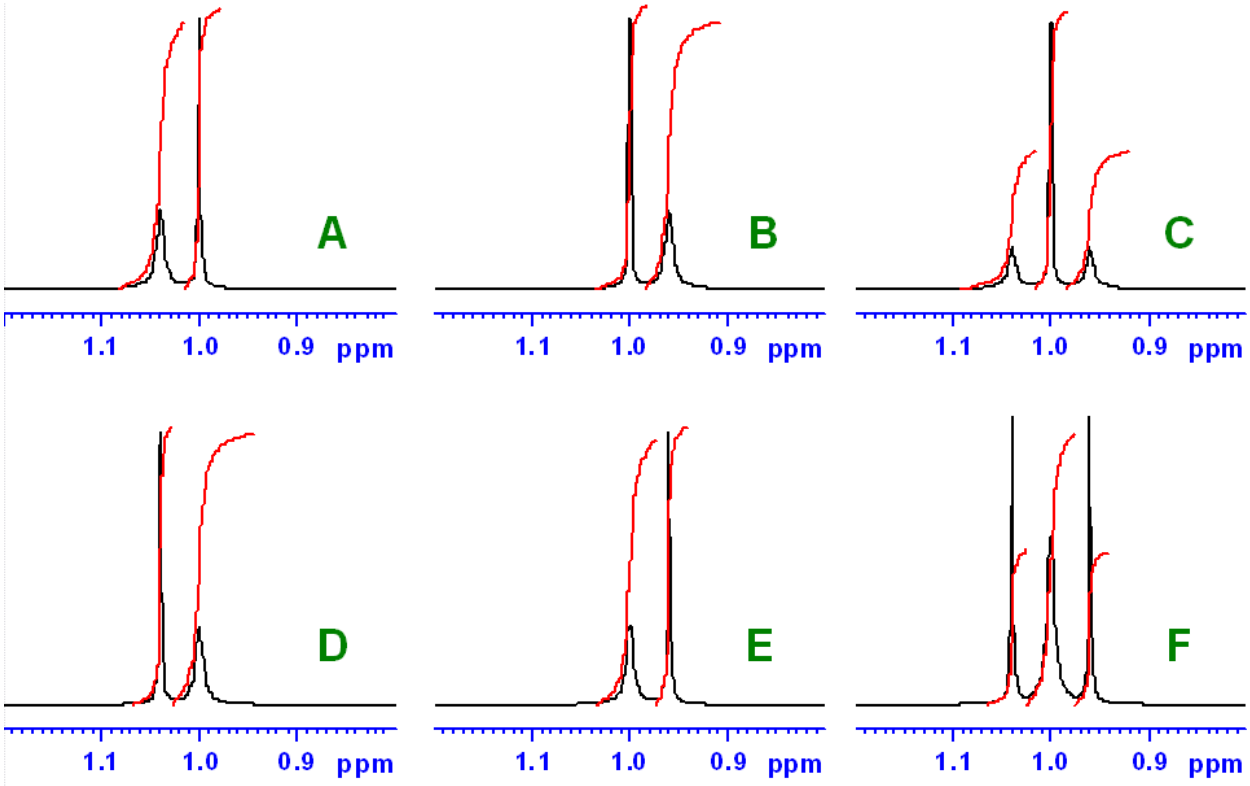
1. The two proton decoupled ^{13}C NMR spectra of a solution of menthol in $CDCl_3$ below were collected on a Varian INOVA 500 NMR spectrometer under the following conditions:
Relaxation delay = 1sec
90 degree pulse used
Acquisition time =1 sec
Number of scans recorded = 1
The only difference between the spectra is that the spectrum on the top was collected with 16 dummy scans in addition to the recorded scan.



(a) What are dummy scans? (2 points)
(b) What is the difference between the two spectra? (2 points)
(c) Why are the two spectra different? (6 points)

2. A spectroscopist obtained the following FID in the "real" channel of an NMR spectrometer:

Which spectrum (or spectra) below would account for this signal? Explain your answer. (10 points)



3. The following ^{1}H 2D NMR pulse sequence was run on a sample using a Varian Unity 500 spectrometer.

D1 - P1 - D0 - P2 - D2 - P1 - ACQUIRE

Some of the parameters are as follows:

The spectral width, SW = 4096 Hz

The relaxation delay, D1 = 1sec

The fixed delay, D2 = 20 msec

Total number of complex points per FID, NP = 2048

Number of increments = 128

The incremented delay, D0 varied linearly with increment number from 0 to 204.8 msec.

The first pulse, P1 = 10 μsec

The second pulse, P2 = 20 μsec

The number of scans for each FID, NS = 16

(a) What is the acquisition time, AQ for each FID? (2 points)

(b) What is the total experiment time? (6 points)

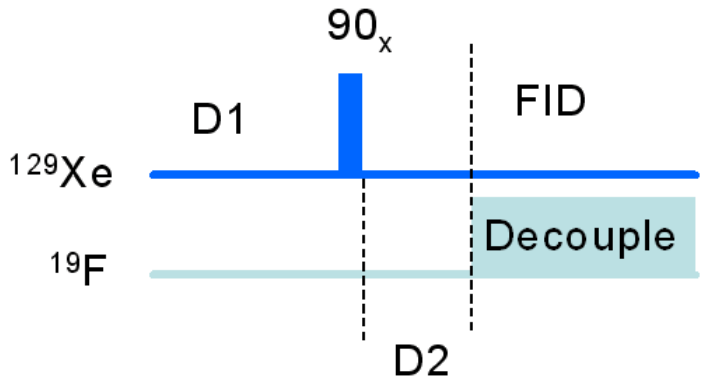
(c) How many Fourier transforms must be calculated to process the data? (4 points)

4. The proton decoupled ^{13}C spectrum of acetone has two peaks at 30 and 207 ppm. A ^{13}C spectrum with ^{1}H decoupling was recorded on a Bruker Avance 400 NMR spectrometer using a spectral width of 20 kHz. The carrier frequency was centred between the peaks.
a) Draw a picture of the spectrum labelling the edges, peaks and centre in ppm. (5 points)
b) The carrier frequency was moved 8 kHz to high frequency and the spectrum was acquired again. Draw a picture of the spectrum labelling the edges, peaks and centre in ppm. (5 points)
c) Redraw the spectra from parts a) and b) labelling the edges, peaks and centre in ppm assuming that digital filtering was used. (4 points)

5. What is the difference in the experimental implementation of heteronuclear vs homonuclear decoupling and why must there be a difference? (8 points)

6. Two NMR resonances with significantly different T_1 relaxation times overlap with one another in a spectrum. Demonstrate how you could obtain subspectra of each resonance. (10 points)

7. XeF_2 was dissolved in a suitable solvent and the following pulse sequence was applied at the ^{129}Xe Larmor frequency.



(a) The carrier was set such that the signal was on resonance. When D2 was set to zero the signal was a positive singlet. As D2 was increased in small increments, the signal became progressively smaller until there was a null signal at D2 = 1.6 msec. What is the value of $^{1}J_{Xe\text{-}F}$? Use vector diagrams to in your answer. (6 points)
(b) If D2 was set at 1.6 msec and the ^{19}F decoupler was not used, what would the spectrum look like? Draw a picture. (6 points)