

1(a) Dummy scans are identical in all respects to any of the other scans using the pulse program except that the receiver is not turned on during the acquisition time.

1(b) The solvent signal is missing from the spectrum which employed 16 dummy scans.

1(c) The T_1 relaxation time for the carbon in deuterated chloroform is much longer than that for the protonated carbons of menthol. The solvent resonance therefore is saturated by the 16 dummy scans and does not show up in the single scan spectrum.

2. The real FID shown consists of a slowly decaying simple exponential and a rapidly decaying cosine function. The slowly decaying exponential represents a sharp line in the middle of the spectrum (on resonance). The rapidly decaying cosine function represents a broad line away from the center of the spectrum (off resonance). Spectra A, B and C all have a sharp on resonance and a broader off-resonance line. Since we do not know what the imaginary FID looks like we cannot distinguish between positive and negative frequencies, all three spectra are possible.

3(a) We know that $SW=(1/DW)$ and $AQ=DW(NP/2)$ therefore $AQ=((NP/2)/SW)$

$$\begin{aligned}AQ &= (2048/2) / (4096 \text{ sec}^{-1}) \\ &= 0.25 \text{ sec}\end{aligned}$$

3(b) Total time = $(D1 + 2P1 + D0 + P2 + D2 + AQ) \cdot (NS) \cdot (\text{number of increments})$

DO varies linearly from the beginning to the end of the experiment so its average can be used in the calculation.

$$\begin{aligned}\text{Total time} &= (1 + 20 \cdot 10^{-6} + 102.4 \cdot 10^{-3} + 20 \cdot 10^{-6} + 20 \cdot 10^{-3} + 0.25) \cdot (16) \cdot (128) \\ &= 2810.76 \text{ sec} \\ &= 46.85 \text{ minutes}\end{aligned}$$

3(c) # transforms needed = # increments + $(NP/2) = 128 + 1024 = 1152$

4(a) Carrier at $(207 \text{ ppm} + 30 \text{ ppm})/2 = 118.5 \text{ ppm}$

SW = 20 kHz need in ppm

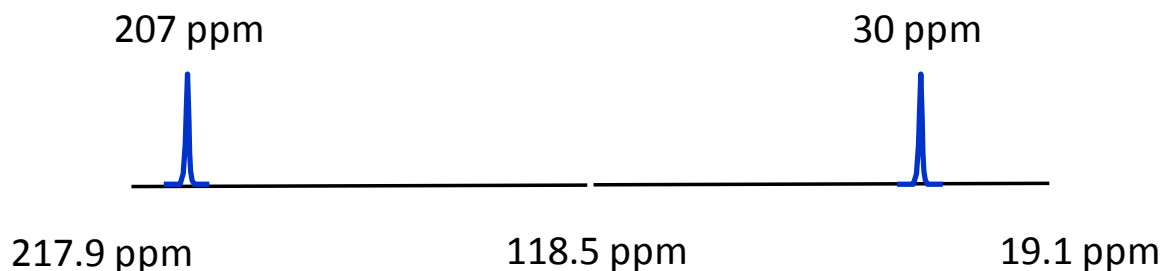
^1H at 400 MHz therefore ^{13}C at 400 MHz ($\gamma_{\text{C}}/\gamma_{\text{H}}$) = 100.60 MHz

1 ppm for $^{13}\text{C} = 100.60 \text{ Hz}$

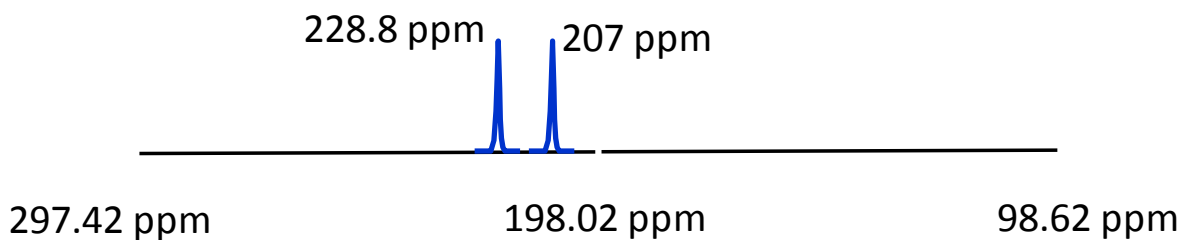
SW in ppm = $20,000 \text{ Hz} / (100.60 \text{ Hz/ppm}) = 198.80 \text{ ppm}$

Window is $118.5 \text{ ppm} \pm (198.8 \text{ ppm} / 2)$

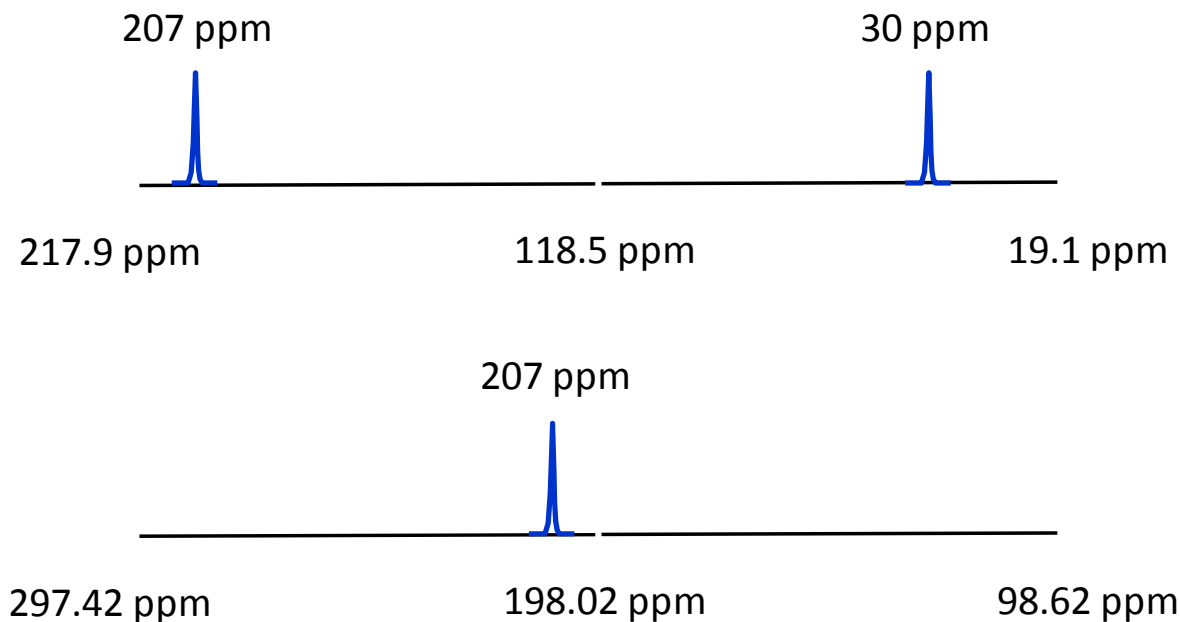
Left limit = 217.9 ppm Right limit = 19.1 ppm



4(b) Carrier moved 8000 Hz to high frequency
 $8000 \text{ Hz} / (100.60 \text{ Hz/ppm}) = 79.52 \text{ ppm}$
 Carrier now at $118.5 \text{ ppm} + 79.72 \text{ ppm} = 198.02 \text{ ppm}$
 New window is $198.02 \text{ ppm} \pm (198.8 \text{ ppm} / 2)$
 Left limit = 297.42 ppm Right limit = 98.62 ppm
 Methyl resonance is outside of the right-hand side of the window by $98.62 - 30 = 68.62 \text{ ppm}$
 It will be folded in on the left-hand side at 68.62 ppm from the left edge
 It will appear at $297.42 - 68.62 = 228.8 \text{ ppm}$

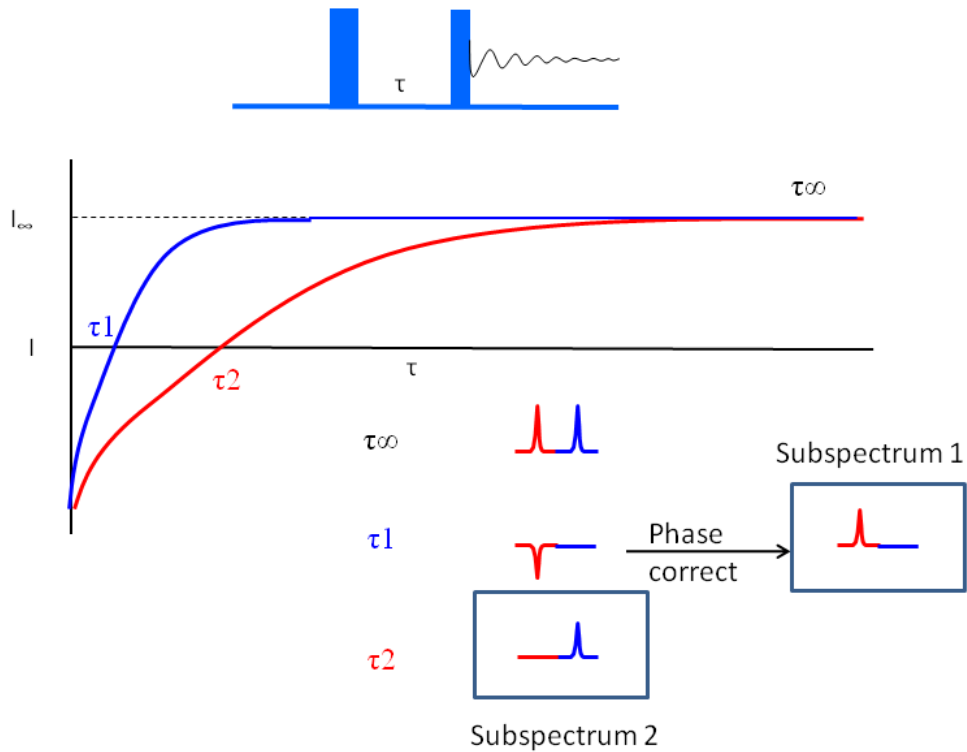


4(c)

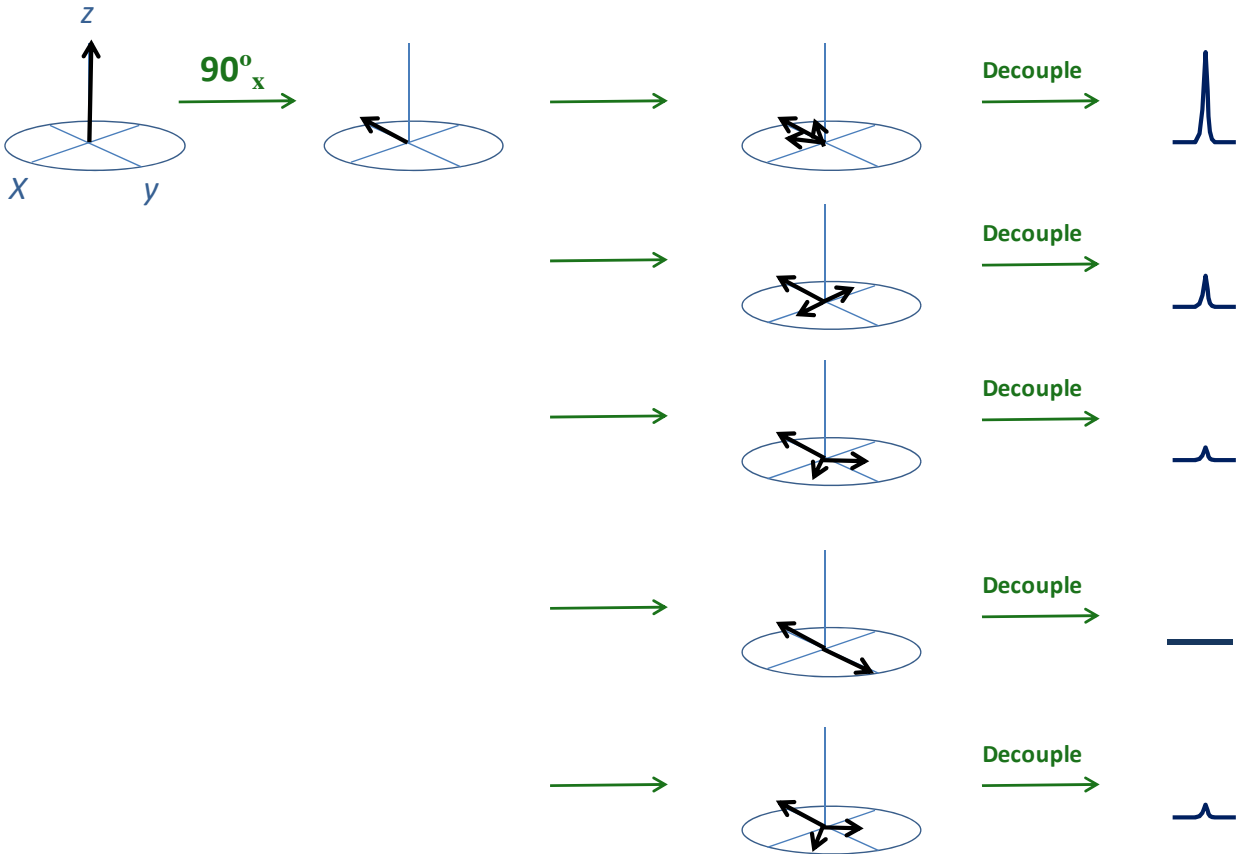


5. Heteronuclear decoupling involves irradiating one type of nucleus while observing another. The decoupling irradiation can be applied during both the dwell period and sampling periods of the FID. Homonuclear decoupling involves irradiating a particular frequency in the spectrum of a particular isotope while observing the rest of the spectrum of the same isotope. It is applied only during the dwell periods of the FID and not during the sampling periods. They are implemented differently because it is usually not possible to simultaneously observe and irradiate a single isotope. It is however possible to irradiate one isotope while observing another.

6. One would apply and inversion recovery sequence setting the recovery delay to a value that would null one of the resonances then repeat the sequence with a recovery delay of a value such that the other resonance was nulled.



7(a) Both ^{129}Xe and ^{19}F are spin $I = \frac{1}{2}$ isotopes.
 In the absence of ^{19}F decoupling the simple ^{129}Xe spectrum would be a 1:2:1 triplet.
 The center-band of the triplet would be in the middle of the spectrum.
 If $D_2 = 0$ the sequence is a simple 1-pulse with decoupling and we would expect a singlet.
 The $^{129}\text{Xe} - ^{19}\text{F}$ coupling constant, $^1J_{\text{XeF}} = X$ Hz. Must find X.
 Follow the sequence with vector diagrams.



After the application of the 90° pulse in the absence of decoupling, the central vector of the triplet remains stationary as it is on-resonance. The outer two vectors rotate at $^1J_{XeF} = X$ Hz. The spectrum is nulled at $D2=1.6$ msec when the two outer vectors of the triplet undergo $\frac{1}{2}$ of a cycle of rotation in the x-y plane. It would therefore take $2*1.6$ msec = 3.2 msec for the vectors to do a full cycle of rotation. This would be $1 \text{ cycle} / 0.0032 \text{ sec} = 312.5 \text{ Hz}$. Therefore $^1J_{XeF} = 312.5 \text{ Hz}$.

7(b) The outer vectors would be 180° out of phase compared to the central vector.

