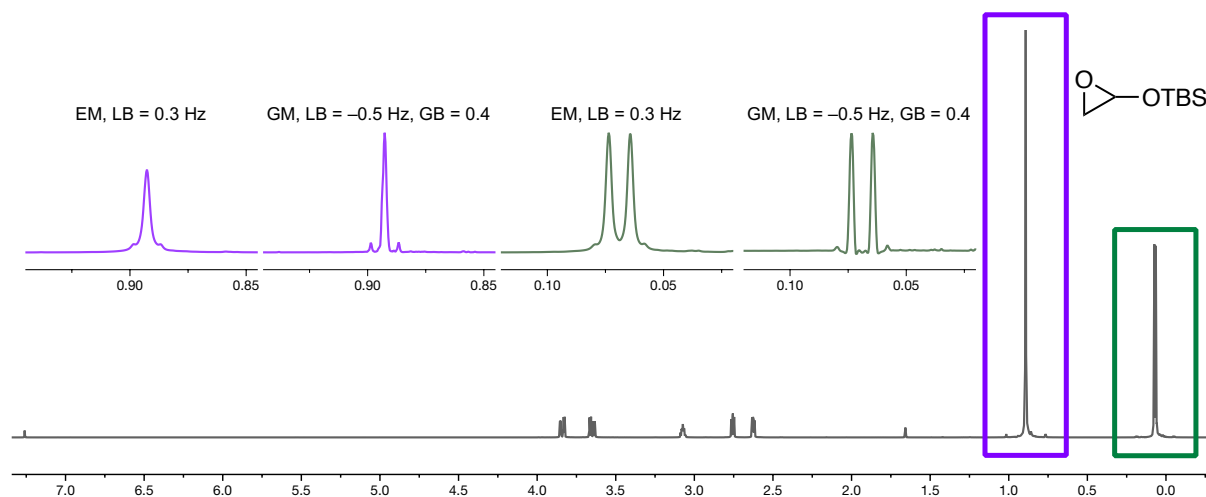## EAA Group Problems – 7 May 2019

1. This ^{1}H spectrum (500 MHz) is from my Part II project. Unfortunately, there seem to be some small "bonus" peaks in the 0–1 ppm region. These are almost invisible with the standard spectral processing (EM), but can be more clearly seen when a Gaussian window function is used (GM). Should I have re-purified the compound? Why or why not?

2. The next ^{13}C spectrum (151 MHz) of a phosphonium salt is kindly provided by Bernie Lee. There should be 10 peaks in the aromatic region; however, only 9 can be seen. Which of the carbons is missing, and why?
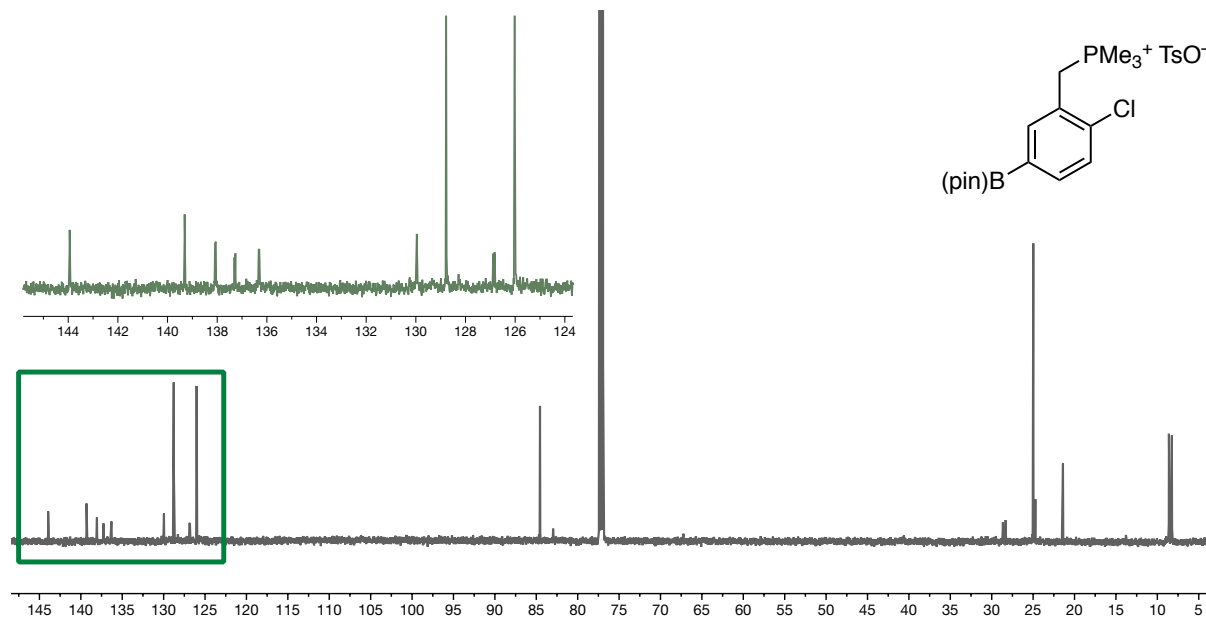
3. The following ¹H spectrum (400 MHz) was magnanimously bequeathed by Steve Mansfield. Asterisks indicate positions which are isotopically labelled with ¹³C. Perplexingly, the benzylic protons (circled in the structure) show up as a triplet, with an apparent coupling of 2.4 Hz. Why is this the case?
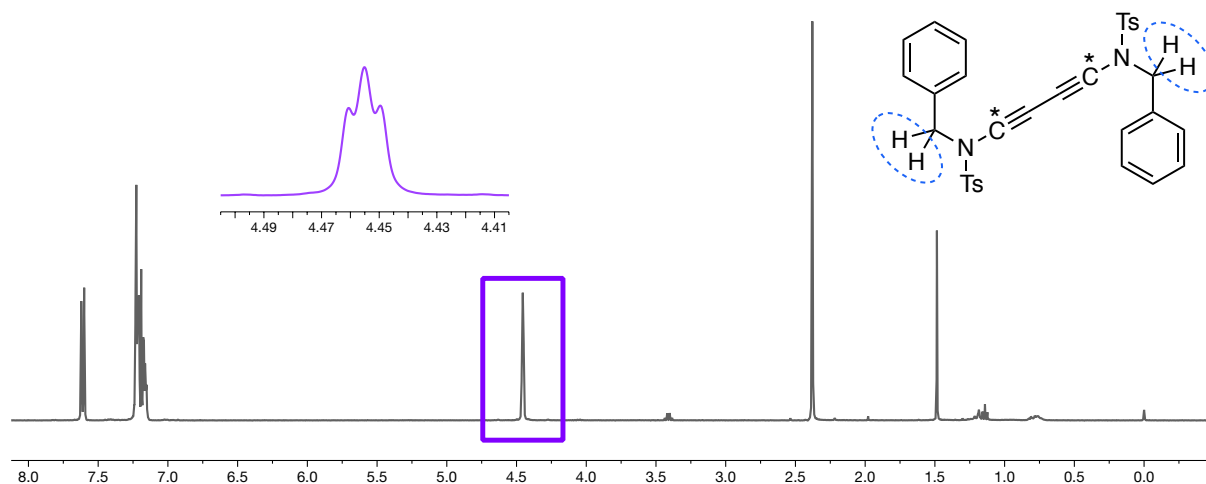
4. For Steve's compound in the previous question, what multiplet pattern would the labelled ¹³C have in the ¹³C spectrum (i.e. is it a singlet, doublet, …)? Don't forget that ¹³C spectra are usually recorded with ¹H decoupling.

5. This ¹H spectrum (600 MHz) was acquired last week as part of my SBM rotation project; the sample itself was kindly provided by Jieyan Lim. The three-bond coupling constant between the two indicated protons is 10.2 Hz. Why is it so large? ($^3J_{HH}$ in acyclic systems are typically in the region of 7 Hz.)
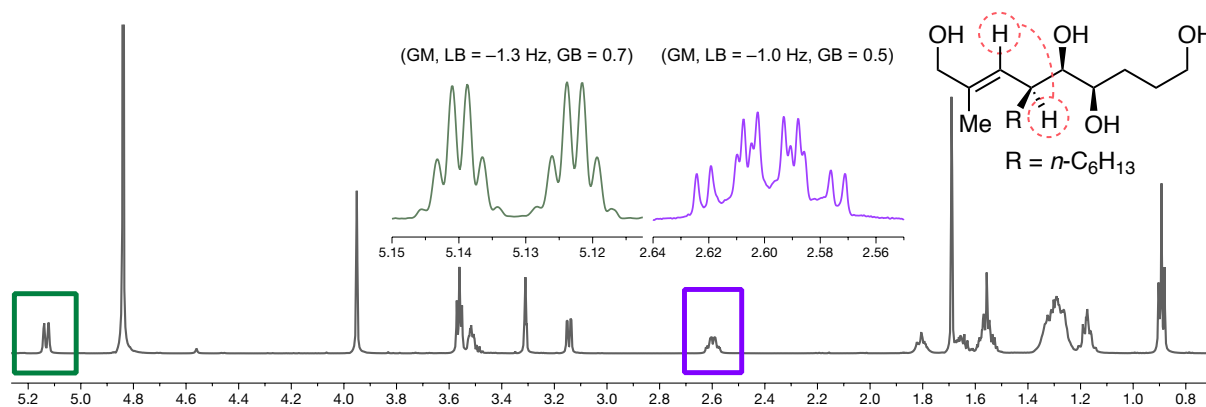
6. The final set of spectra (400 MHz ^{1}H, 101 MHz ^{13}C) are again taken from my Part II project. Neglecting some roofing, the aromatic region of the ^{1}H is a nice first-order spectrum (i.e. H2 and H5 are clean dd's, whereas H3 and H4 are clean td's). But it is not possible to decide which dd is H2 and which is H5, even with the help of 2D spectra… or is it?

How would you choose between the two possible assignments, using all the information below (obtained from COSY, HSQC, and HMBC spectra)? You're allowed to "run" other experiments if you think that they would give you useful information!

Peak description	Assignment A	Assignment B
132.0 ppm	C1 (SO$_2$NRR')	C6 (NO$_2$)
8.10 ppm (dd, J = 7.8, 1.6 Hz)	H2	H5
131.80 ppm	C2	C5
7.70 ppm (td, J = 7.7, 1.6 Hz)	H3	H4
131.82 ppm	C3	C4
7.75 ppm (td, J = 7.6, 1.6 Hz)	H4	H3
134.6 ppm	C4	C3
7.64 ppm (dd, J = 7.7, 1.6 Hz)	H5	H2
124.3 ppm	C5	C2
148.6 ppm	C6 (NO$_2$)	C1 (SO$_2$NRR')