Chemistry 5326: Organic Spectroscopic Analysis
Fall 2015
Group Project

The whole class will be assigned a set of spectra for an unknown compound. You are to work in groups of three that will be posted on the class website. The data supplied will be IR, MS, ^{1}H, ^{13}C, and a number of 2D NMR spectra. You are to reach a group decision as to what the structure is. While determining absolute stereochemistry may not be needed or possible, any relative stereochemistry should be determined to the best of your ability. Remember, the spectra that you are analyzing are real data. As such, there may be a few extraneous peaks you will have to rule out.

Available data: The NMR data will be sent via email as a ZIP file containing several files. If you want access to different views of the data let me know. All spectra should be turned in with your final report.

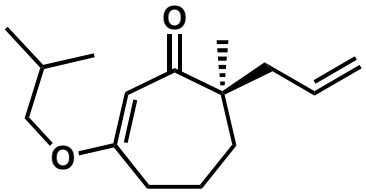
Report Format:
The final report will be submitted in *Organic Letters* format. With all individual's names (you do not need to assign a "starred" author). The templates and instructions for working with them are available on the ACS website:

> *Organic Letters* website → Submission & Review → Information for Authors → Document Templates

This final report should give your final deduced structure and describe how you went about solving the structure. Even though the unknown is a purely synthetic compound, treat the report as if you were isolating a natural product. It will be helpful to look through a few natural product isolation papers from *Organic Letters, Journal of Organic Chemistry,* and/or *Journal of Natural Products* to see what sort of information should be included. In general you should include some background information as to where and when the "sample was collected" (be creative) along with a written explanation as to how you arrived at your final structures. You should also provide assignments for as many ^{13}C and ^{1}H signals as possible. Feel free to include "biological data" if you want (again, be creative). There are examples from previous years posted on the class website. You do not need to include literature references, but you should have footnotes that include the spectral data in the correct format (see below).

For the ^{1}H NMR data it is acceptable to use the following abbreviations: app = apparent, br = broad, obsc = obscured, bs = broad singlet. The use of "m" is *ONLY* acceptable in the following cases: 1) multiple peaks are overlapping to such an extent that they cannot be distinguished and/or *J* values cannot be calculated, 2) the peak is truly a multiplet (e.g., non-first order splitting pattern, multiple overlapping peaks that cannot be distinguished). It is *NOT ACCETPABLE* to substitute "m" for ddddd or similarly complicated splitting patterns. I have calculated *J* values for a ddddd….you can too. For ^{13}C, all that is needed is the chemical shifts of the carbons. For IR, all that is needed are the frequencies of the IR peaks that represent the important functional groups. The IR spectra will say what the sample conditions were (i.e. Nujol, NaCl plate, KBr pellet, thin film, etc.) if it does not, assume it was a NaCl plate as a thin film. If you need guidance putting this information together or working with the template feel free to ask me (but I will not be able to tell you if your structure is correct).

Example writeup:



^{1}H NMR (300 MHz, $CDCl_3$) δ 5.72 (dddd, *J* = 7.2, 7.2, 10.8, 15.9 Hz, 1H), 5.31 (s, 1H), 5.05 (br s, 1H), 5.01 (dddd, *J* = 1.5, 1.5, 2.7, 5.1 Hz, 1H), 3.51 (dd, *J* = 6.6, 9.3 Hz, 1H), 3.45 (dd, *J* = 6.6, 9.3 Hz, 1H), 2.50-2.45 (m, 2H), 2.38 (app dd, *J* = 7.2, 13.5 Hz, 1H), 2.20 (dddd, *J* = 1.2, 1.2, 7.5, 8.7 Hz, 1H), 1.98 (app sept, *J* = 6.6 Hz, 1H), 1.89-1.70 (m, 3H), 1.63-1.55 (m, 1H), 1.14 (s, 3H), 0.95 (app d, *J* = 6.9 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 206.5, 171.1, 134.4, 117.7, 104.8, 74.3, 51.3, 45.2, 35.9, 35.0, 27.8, 25.0, 19.7, 19.1; IR (Neat Film NaCl) 3075, 2959, 2932, 1614, 1470, 1387, 1213, 1192, 1172, 998, 912 cm^{-1}; HRMS (EI) 236.1776 calc'd for $C_{15}H_{24}O_2$, found 236.1775.

If for some reason you are unable to give a complete structure, provide as many substructures as possible along with the relevant spectral evidence.