Week 1: Spectroscopic Identification of an Unknown

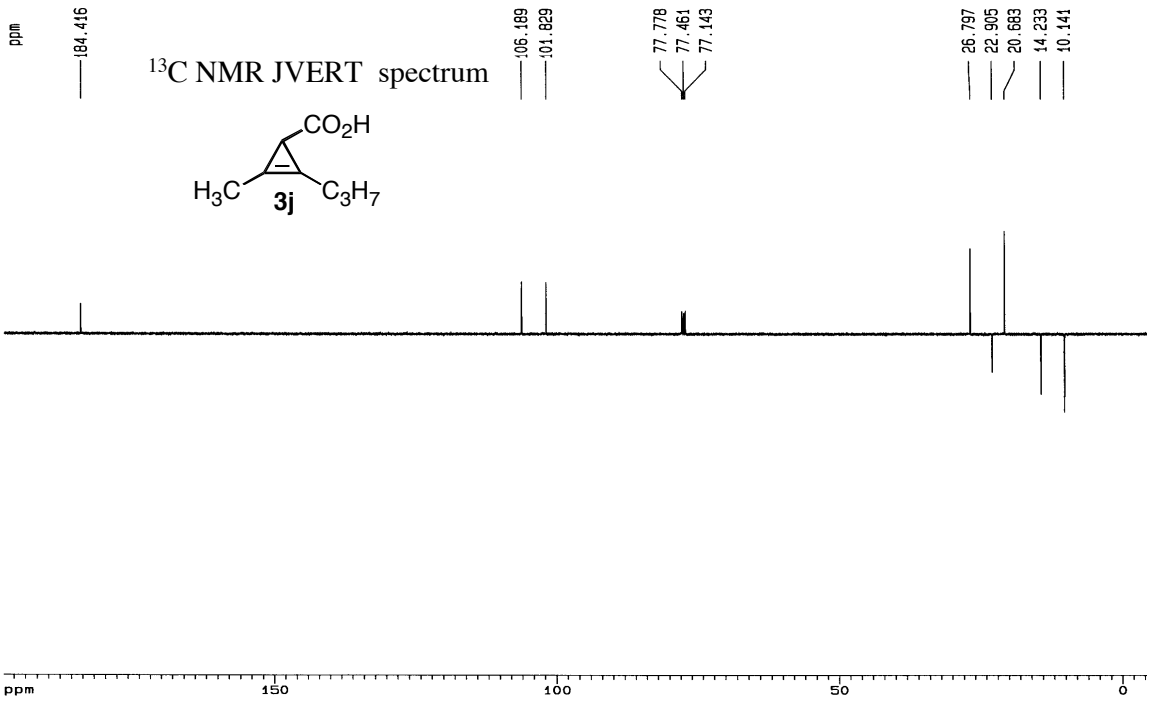
This weeks assignment is very simple: you get a vial with a molecular formula. Tell us what it is. Just like CHEM333, but you provide the data!

In lab, you will receive a substance, and you will perform ^{1}H NMR, ^{13}C NMR and IR spectroscopy. Your TA will first show you how to prepare samples.

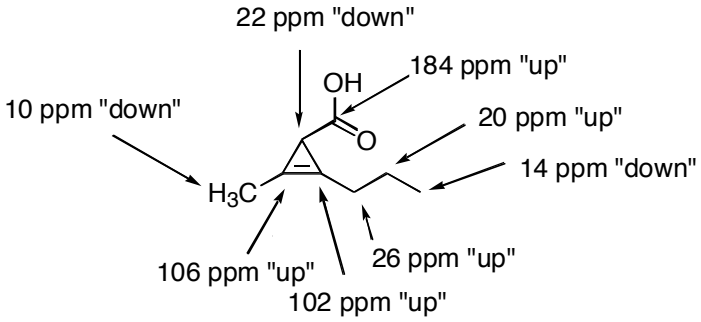
You will submit an NMR tube to be analyzed during the week; data will be sent to you. Collect the IR during lab

The hard work is outside of lab this week in the assignment of your structure and in the preparation of your lab report. Your report will simply consist of a structure assignment, and your rationale for that assignment using ^{13}C NMR, ^{1}H NMR and IR.

^{13}C NMR. In CHEM333, all of your resonances for ^{13}C NMR were given with a multiplicity of singlet (s), doublet (d), triplet (t) or quartet (q). In reality, it is not usually practical to acquire spectra with direct coupling to protons because the peaks tend to overlap. Instead, we usually obtain information about multiplicity by acquiring a ^{13}C "JVERT" spectrum. In ^{13}C JVERT, methylenes and quaternary carbons appear "up" (with the same phase as the $CDCl_3$ triplet), and methyl and methine carbons appear "down". In otherwords, peaks that you normally see assigned as "s" or "t" appear "up", whereas peaks that you normally see assigned as "d" or "q" appear "down". Consider the spectrum of compound **3j** below:



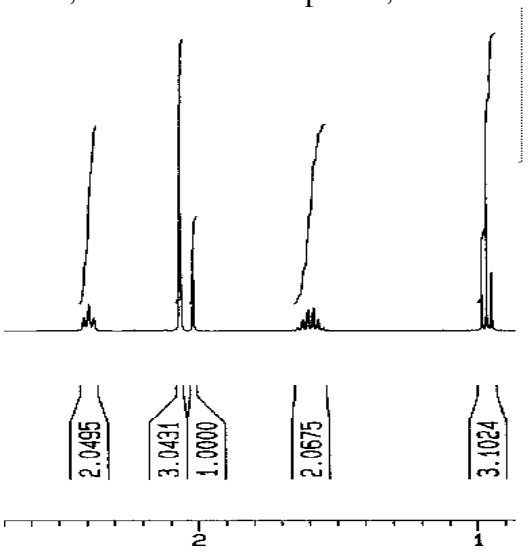
Here is an assignment of the resonances for this compound:



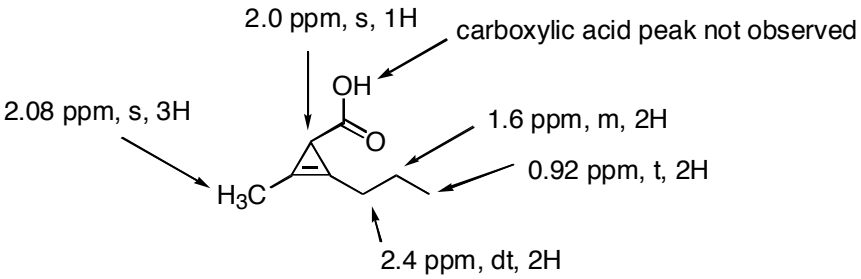
Your lab report should have a diagram similar to the one shown above for the assignment of chemical shifts!

^{1}H NMR.

Likewise, your lab report should have an assignment of peaks in the ^{1}H NMR spectrum. Thus, for the same compound, we observe the following ^{1}H NMR spectrum

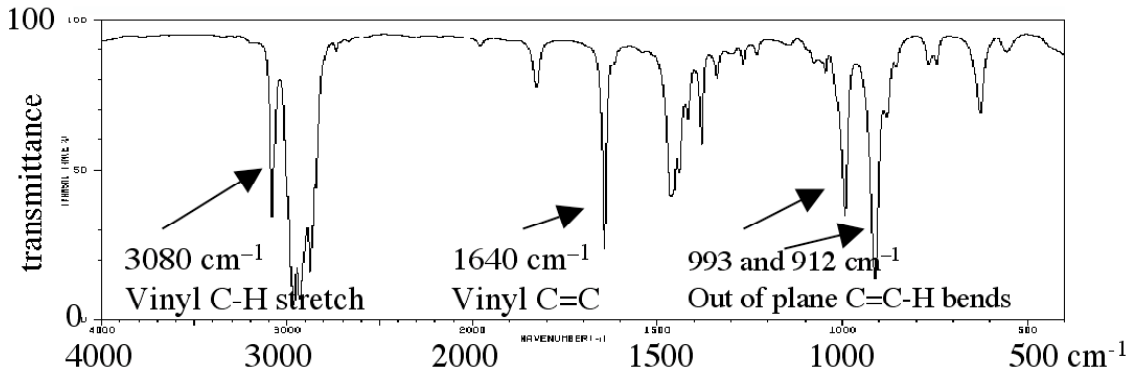


Your report should contain a diagram of this type to explain the chemical shifts:



IR spectroscopy

Your report should also assignment those peaks that are associated with the major functional groups in the molecule. For example, for 1-pentene



Table

3080 cm^{-1}:	vinyl C-H stretch
1640 cm^{-1}:	vinyl C=C stretch
993 cm^{-1}:	out of plane C=C-H bend
912 cm^{-1}:	out of plane C=C-H bend

Points will be deducted for missed assignments of major functional groups!