344 Organic Chemistry Laboratory

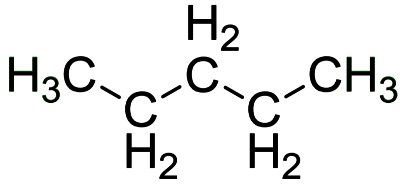
Introduction to ^{13}C-NMR Spectroscopy

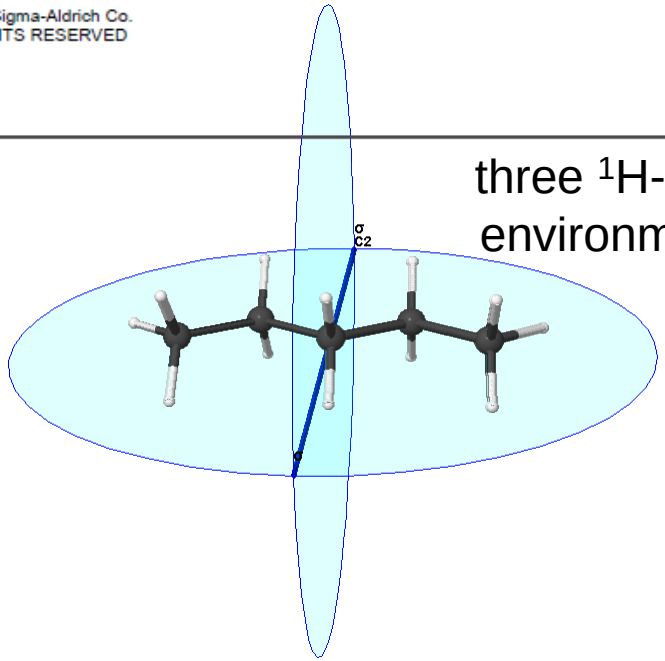
Main topics

- ^{13}C-atom chemical shift range

-^{1}H-coupled and decoupled ^{13}C-NMR spectra

^{1}H-NMR Spectrum of *n*-pentane





300 MHz ^{1}H NMR
In CDCl3

three ^{1}H-atom environments

6.00

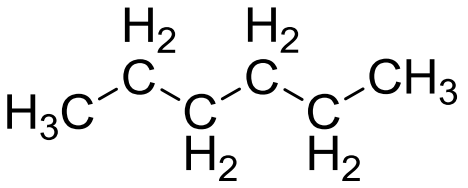
5.94

TMS

2.0 1.8 1.6 1.4 1.2 1.0 0.8 0.6 0.4 0.2 -0.0

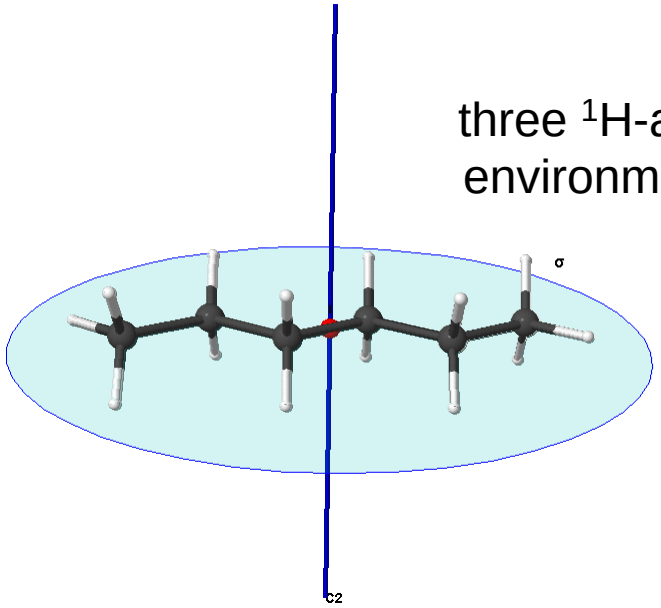
2

^{1}H-NMR Spectrum of *n*-hexane



300 MHz ^{1}H NMR
In CDCl3

three ^{1}H-atom environments

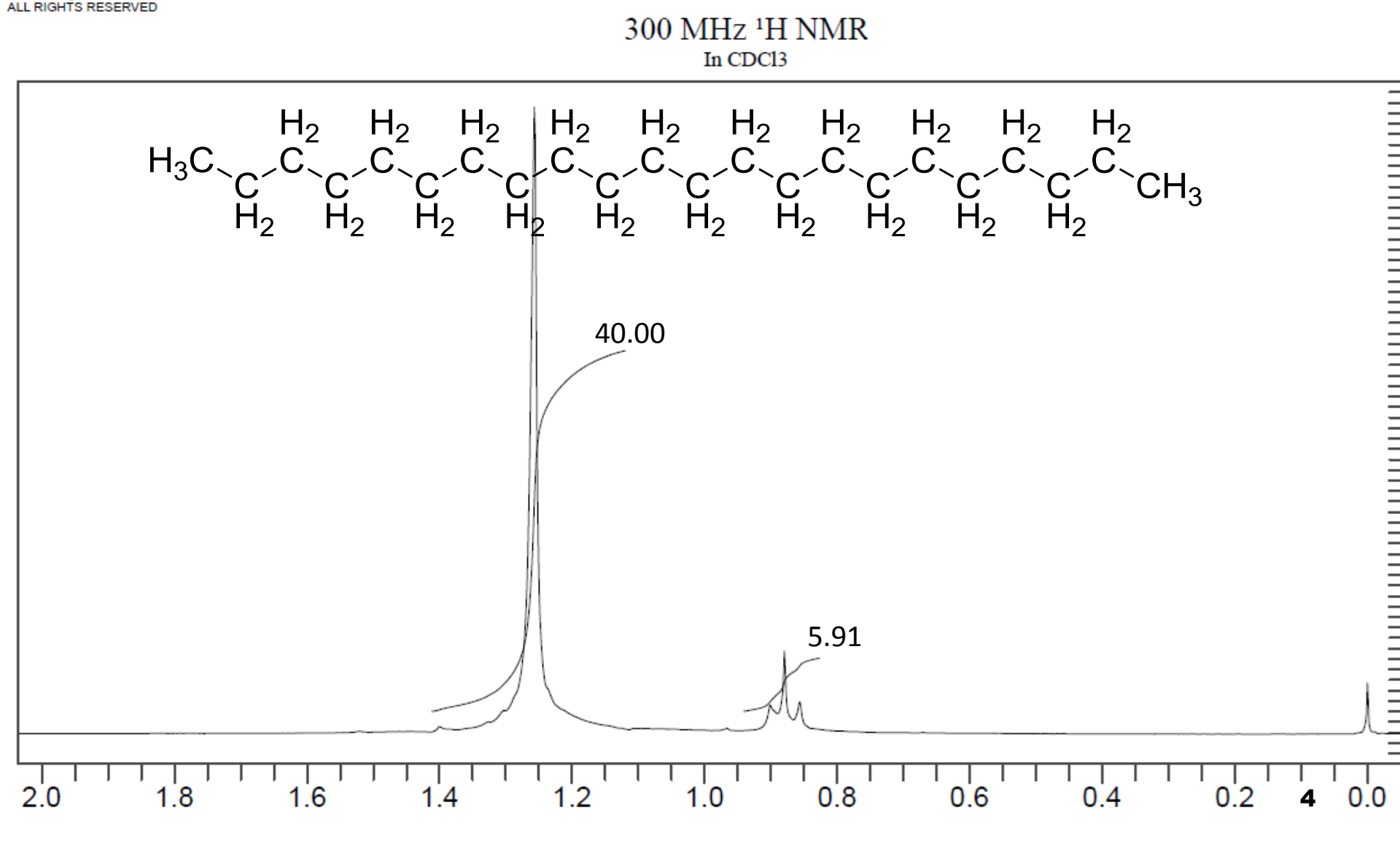


8.00

6.00

2.0 1.8 1.6 1.4 1.2 1.0 0.8 0.6 0.4 0.2 3 -0.0

^{1}H-NMR Spectrum of *n*-docosane

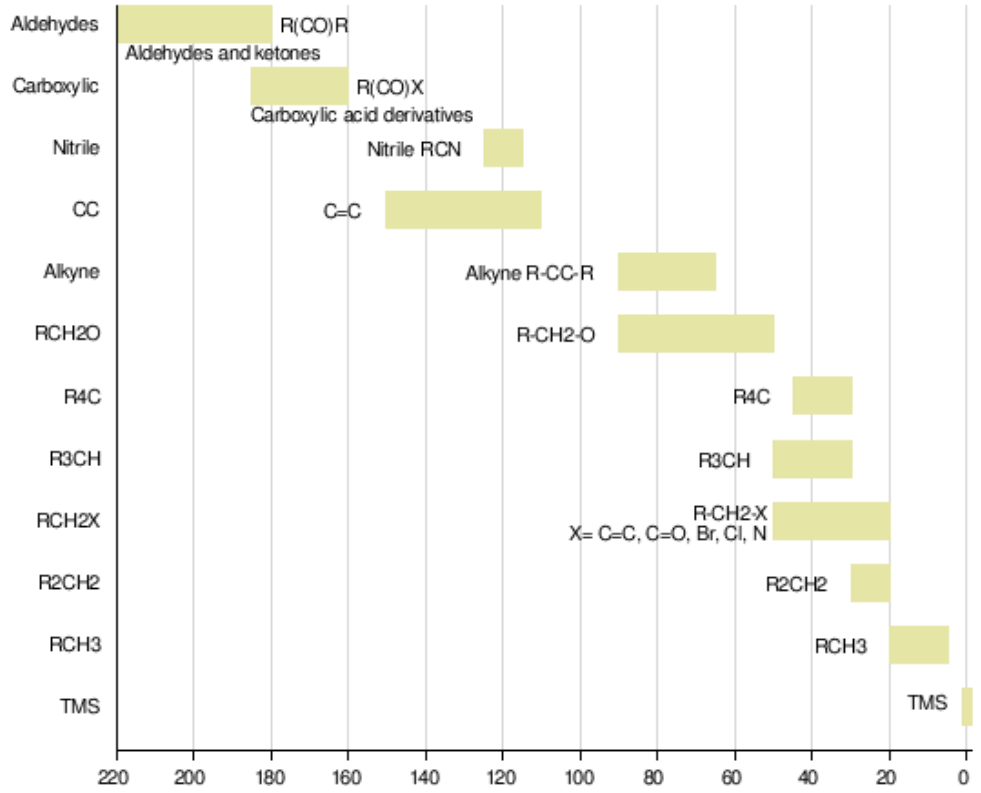


^{13}C-NMR Spectroscopy

^{13}C is NMR active (I = ½); ^{12}C is NMR inactive (I = 0).

The natural abundance of ^{13}C is ~1.1%.

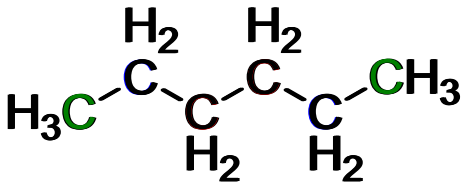
A greater chemical shift range provides greater better differentiation of signals; reduced 2nd order effects



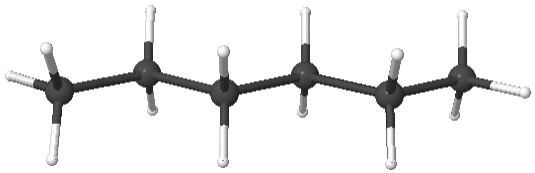
(~220 ppm)

Often the NMR experiment is performed in a ^{1}H-decoupled manner to simplify the spectrum; removes coupling to H-atoms.

^{13}C-NMR Spectrum of *n*-Hexane



75 MHz ^{13}C NMR
In CDCl3

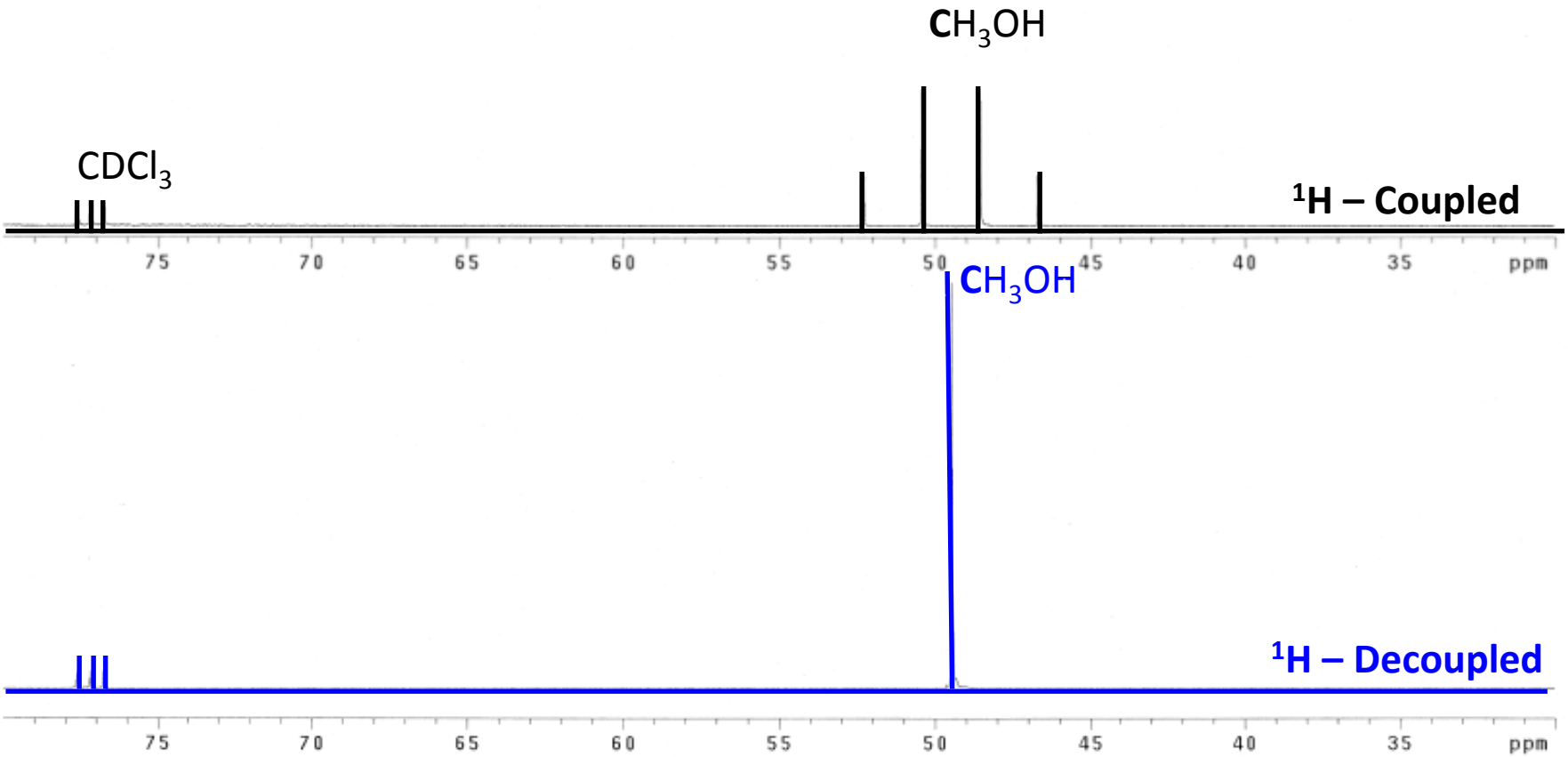


three ^{13}C-atom environments

^{13}C-NMR Spectrum of Methanol

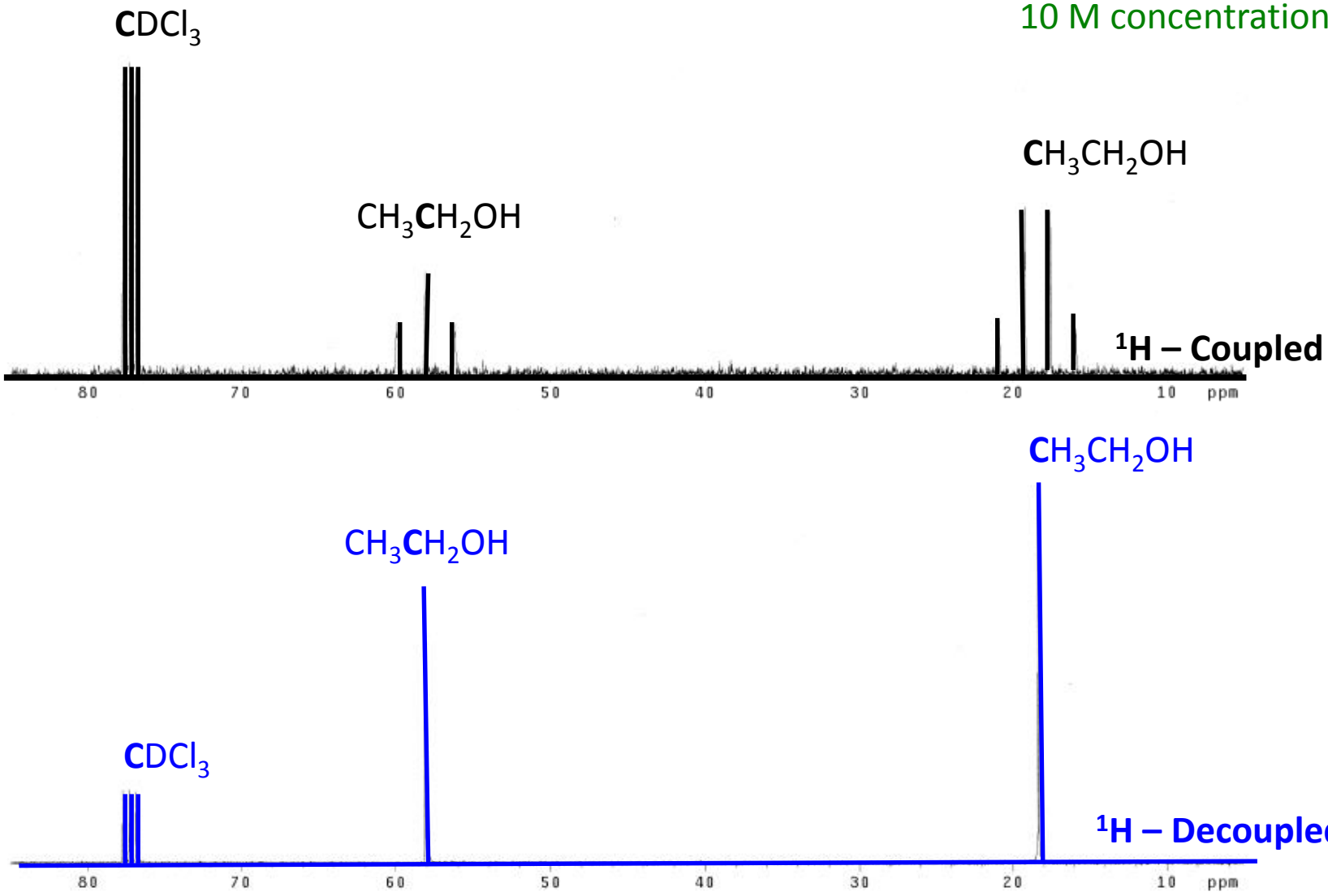
512 scans
30 min
10 M concentration

^{1}H is > 99% abundant; it couples strongly to ^{13}C –atom it is attached to ($^{1}J_{HC}$ = 100-210 Hz) with normal *n+1 rule* splitting.

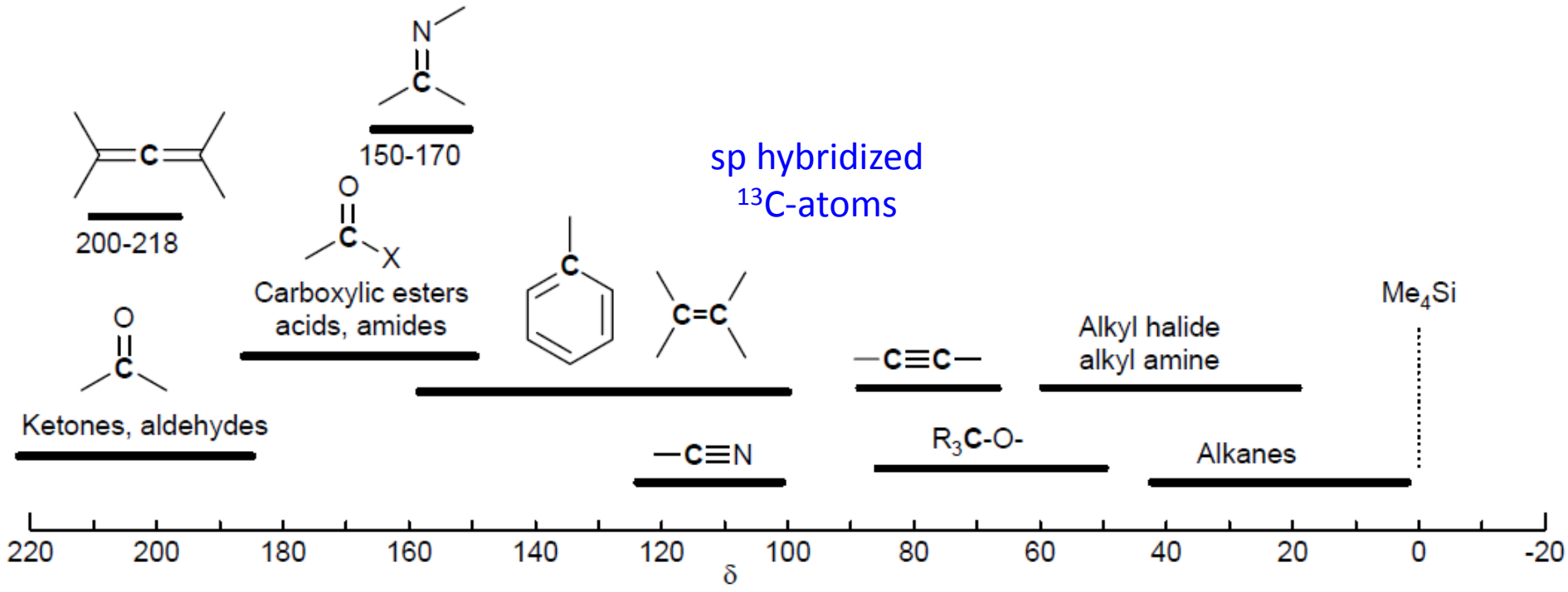


^{13}C-NMR Spectrum of Ethanol

512 scans
30 min
10 M concentration



^{13}C-NMR Spectroscopy Chemical Shift Ranges



sp hybridized ^{13}C-atoms

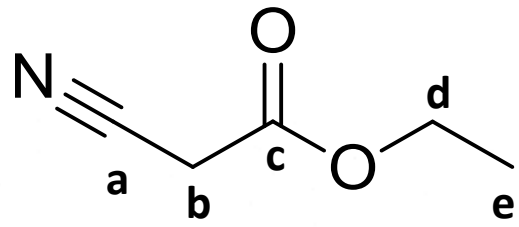
sp^2 hybridized ^{13}C-atoms

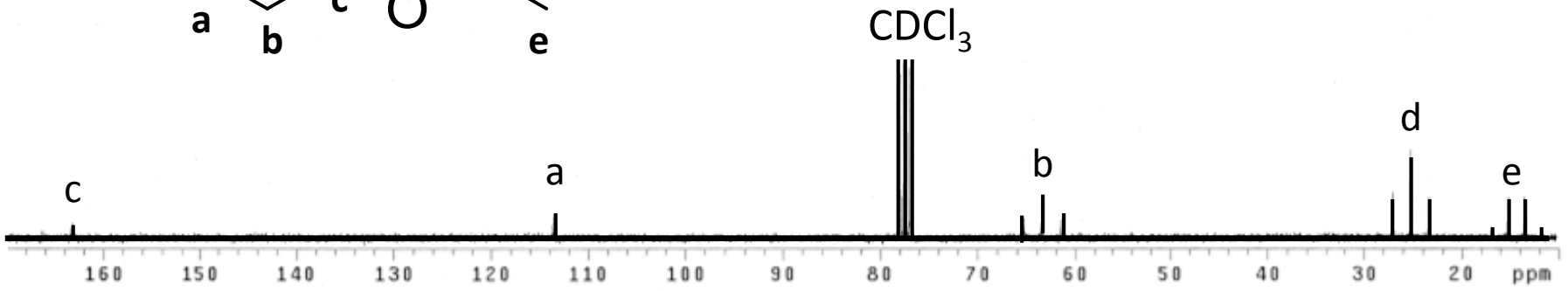
sp^3 hybridized ^{13}C-atoms

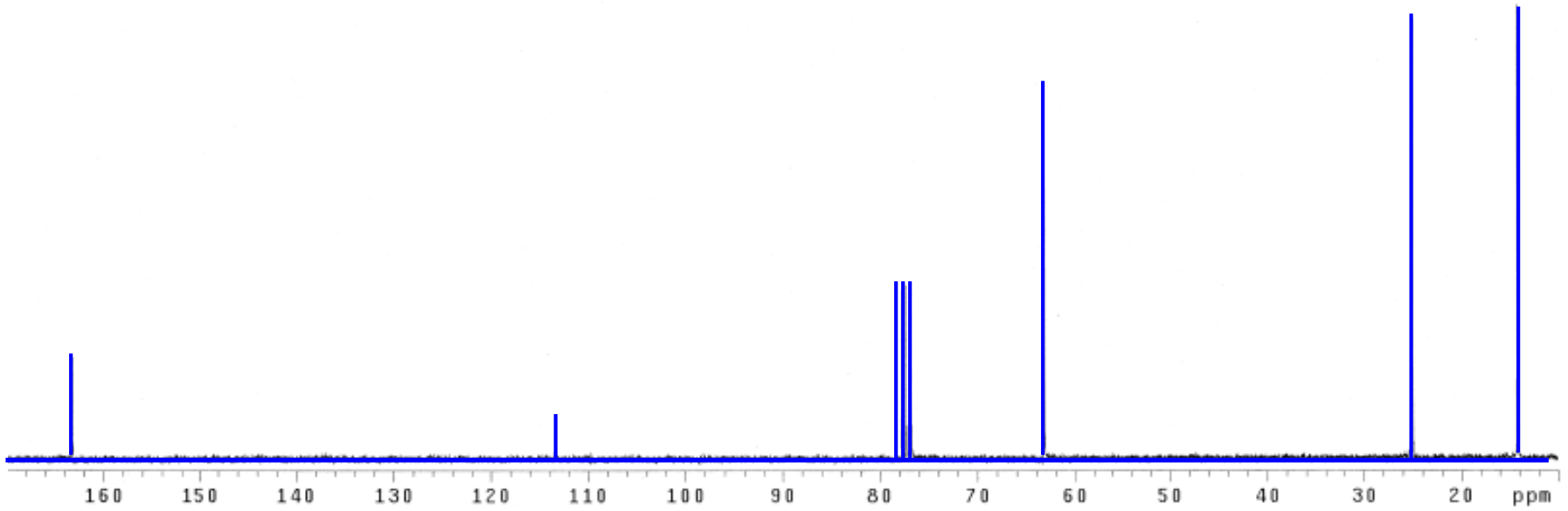
Reich, Hans J. http://www.chem.wisc.edu/areas/reich/handouts/chem343-345/345-nmr-handout.pdf

^{13}C-NMR Spectrum of Ethyl Cyanoacetate

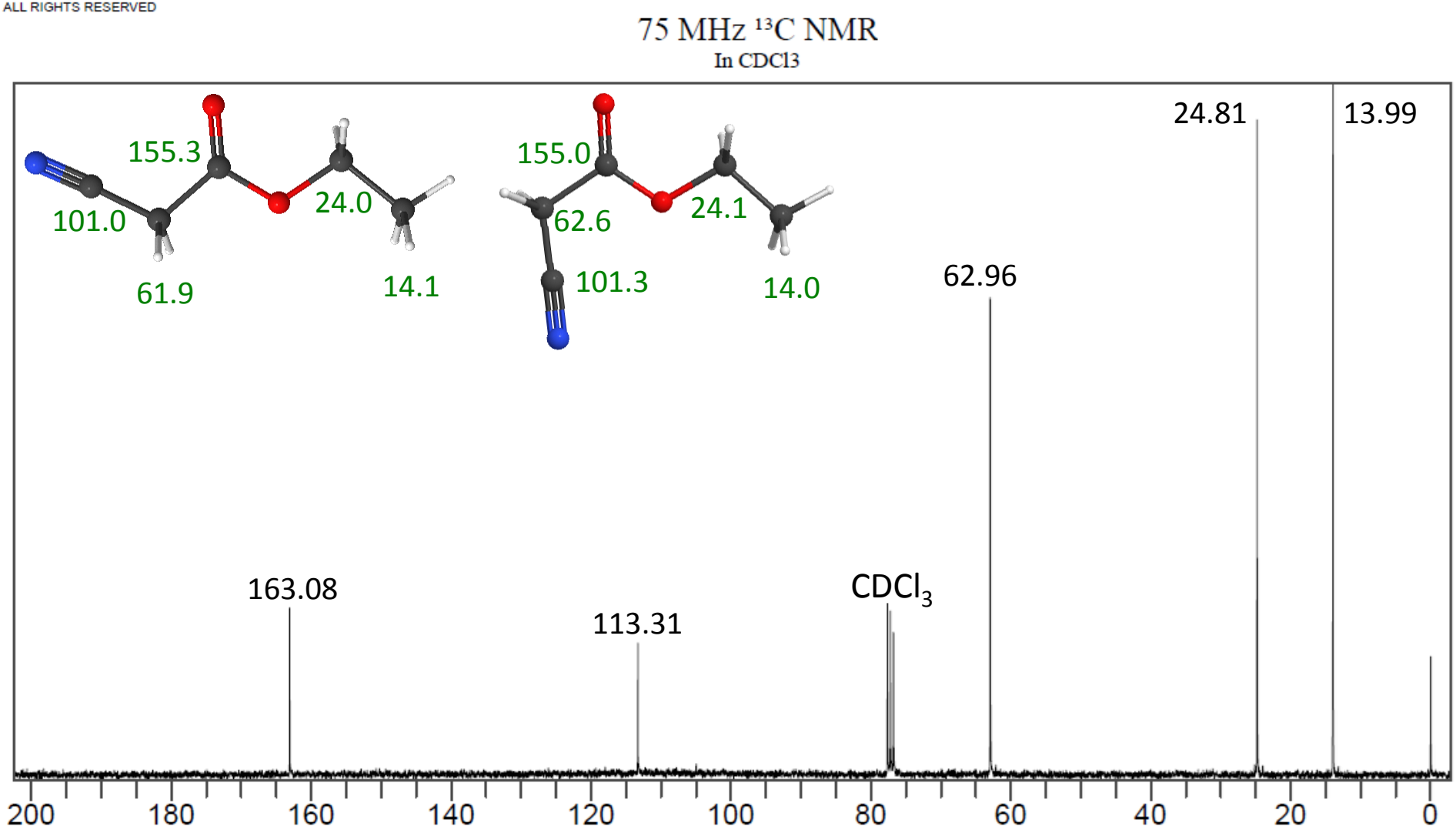
512 scans
30 min
0.35 M concentration





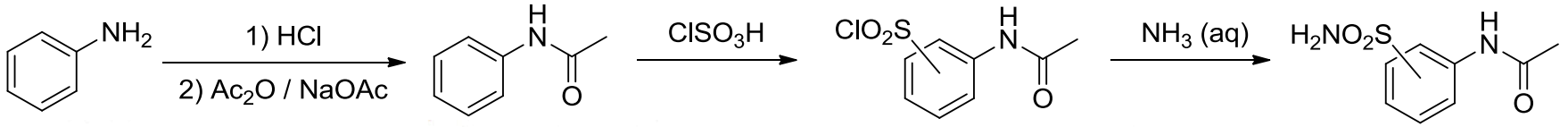


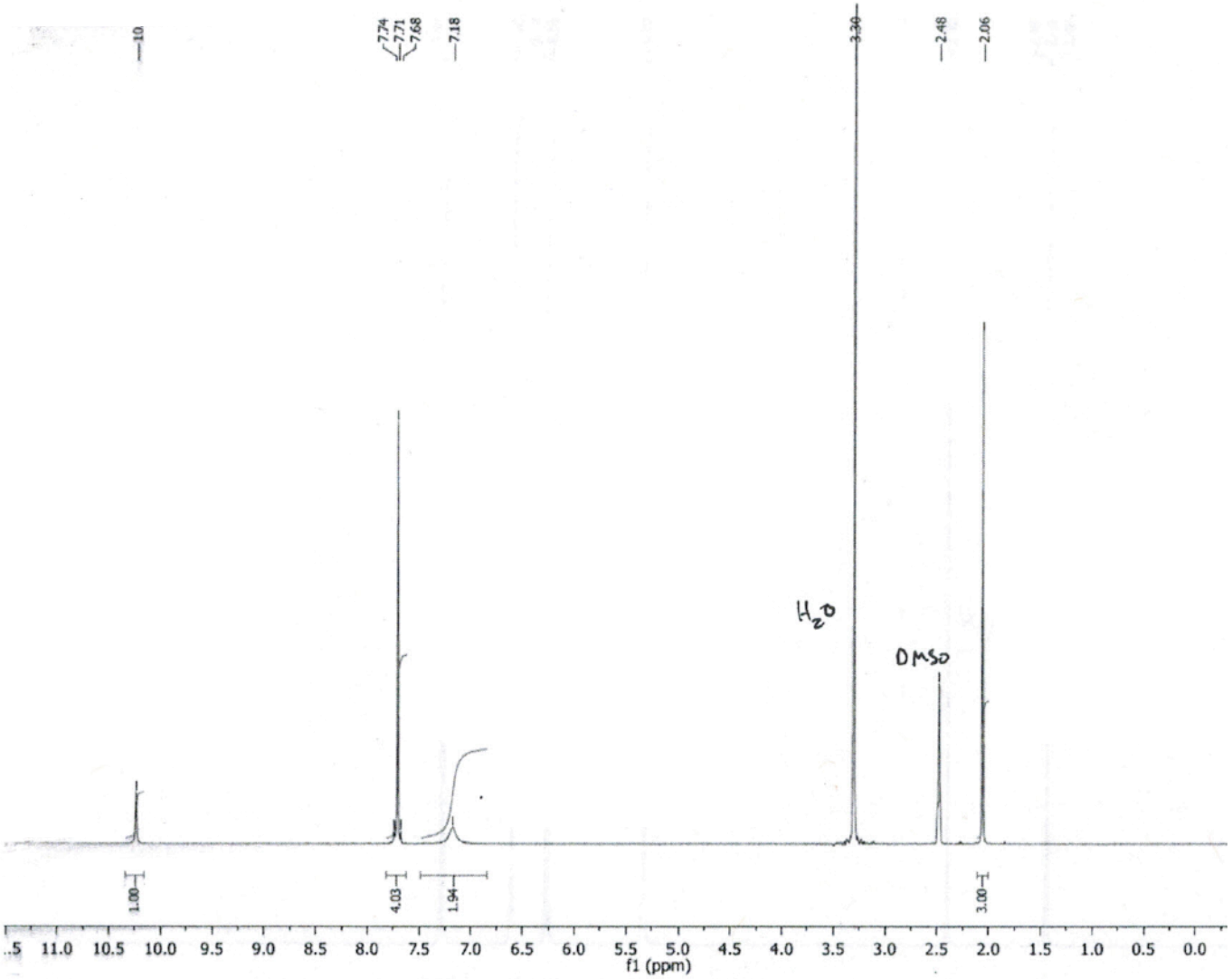
^{13}C-NMR Spectrum of Ethyl Cyanoacetate

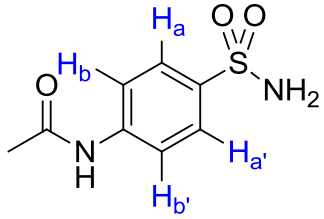


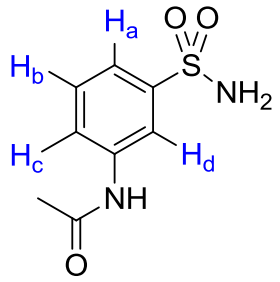
Isotropic NMR Shifts relative to TMS calculated with WebMO/Gaussian09 at B3LYP/6-31G(d)

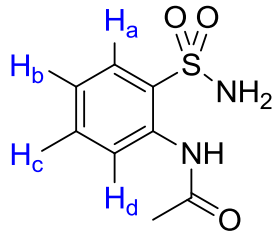
Determination of 4’-sulfamoylacetanilide Regiochemistry











Determination of 4’-sulfamoylacetanilide Regiochemistry