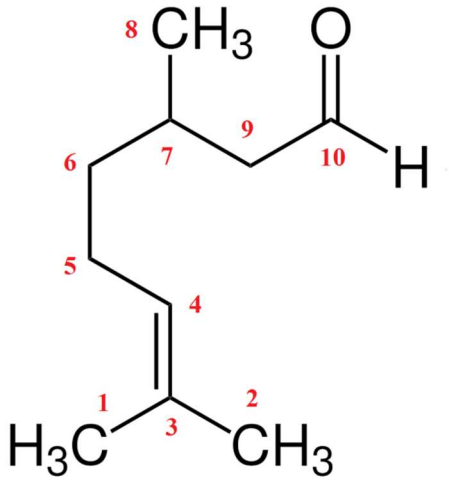
Citronellal



Assignment	^{1}H Chemical Shift	^{13}C Chemical Shift
1 or 2	1.600 (singlet)	17.175
1 or 2	1.675 (singlet)	25.253
3	n/a	131.058
4	5.086	123.803
5	1.8-2.1	25.052 or 27.636
6	1.32-1.45	36.587
7	1.8.-2.1	25.052 or 27.636
8	0.962 (doublet)	19.446
9	2.1-2.4	50.594
10	9.731 (triplet)	201.895

Assignments

It is possible to make most of the assignments in the spectrum. Some are obvious and can be assigned on the basis of chemical shift and multiplicity; others require using a combination of the HETCOR and COSY experiments.

Assignment of C10/H10 and C9/H9

The carbon signal at 201.895 clearly belongs to the carbonyl carbon, C10. In the HETCOR this shows a correlation to a triplet at 9.731 ppm, allowing it to be assigned to H10 (which could have been deduced from chemical shift alone.)

H10 can only be split by H9. Consulting the COSY, the signal at 9.731 shows cross peaks to a region of the proton spectrum at 2.1 – 2.2 ppm, allowing it to be assigned to the H9 hydrogen atoms. This region of the spectrum correlates to the carbon signal at 50.594 ppm, allowing this to be assigned to C9.

Assignment of C3 and C4/H4

Only two carbon signals appear within the alkene region, at 131.058 and 123.803 ppm. The signal at 131.058 does not show any correlations in the HETCOR and can be assigned to C3; this leaves the signal at 123.803 to be assigned to C4. This shows a correlation to a proton triplet at 5.086, allowing it to be assigned to H4.

Assignment of C1/H1 and C2/H2

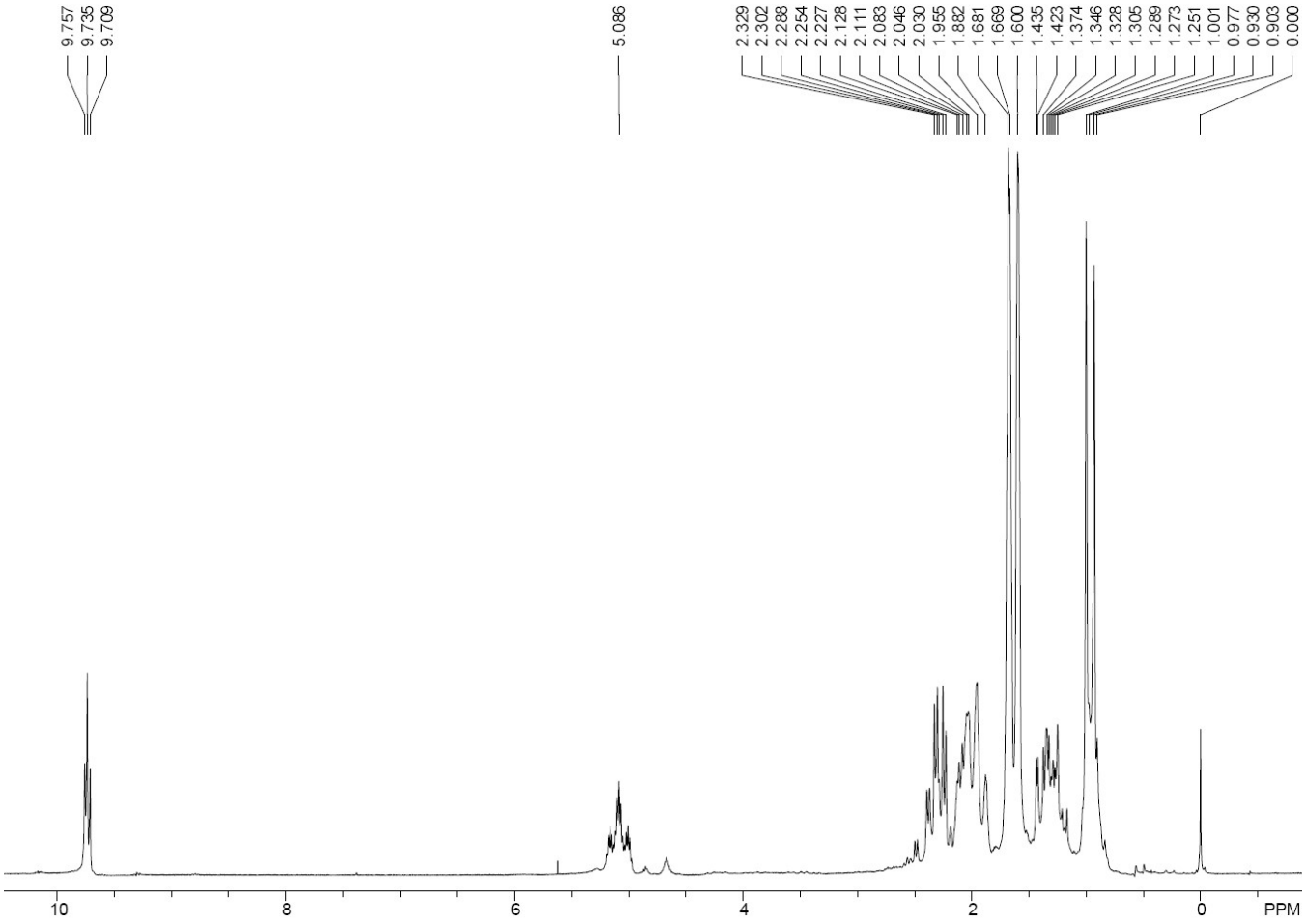
In the COSY spectrum, the proton triplet at 5.086 (H4) shows very strong correlations to the singlets at 1.600 and 1.675 ppm. These correlate to carbon signals at 17.175 and 25.253, respectively, but it cannot be determined which is C1/H1 and which is C2/H2

Note that such couplings take place over four bonds. In fact, the signal at 1.675 ppm appears split into two peaks at 1.669 and 1.681, which suggests it may not be a “real” singlet.

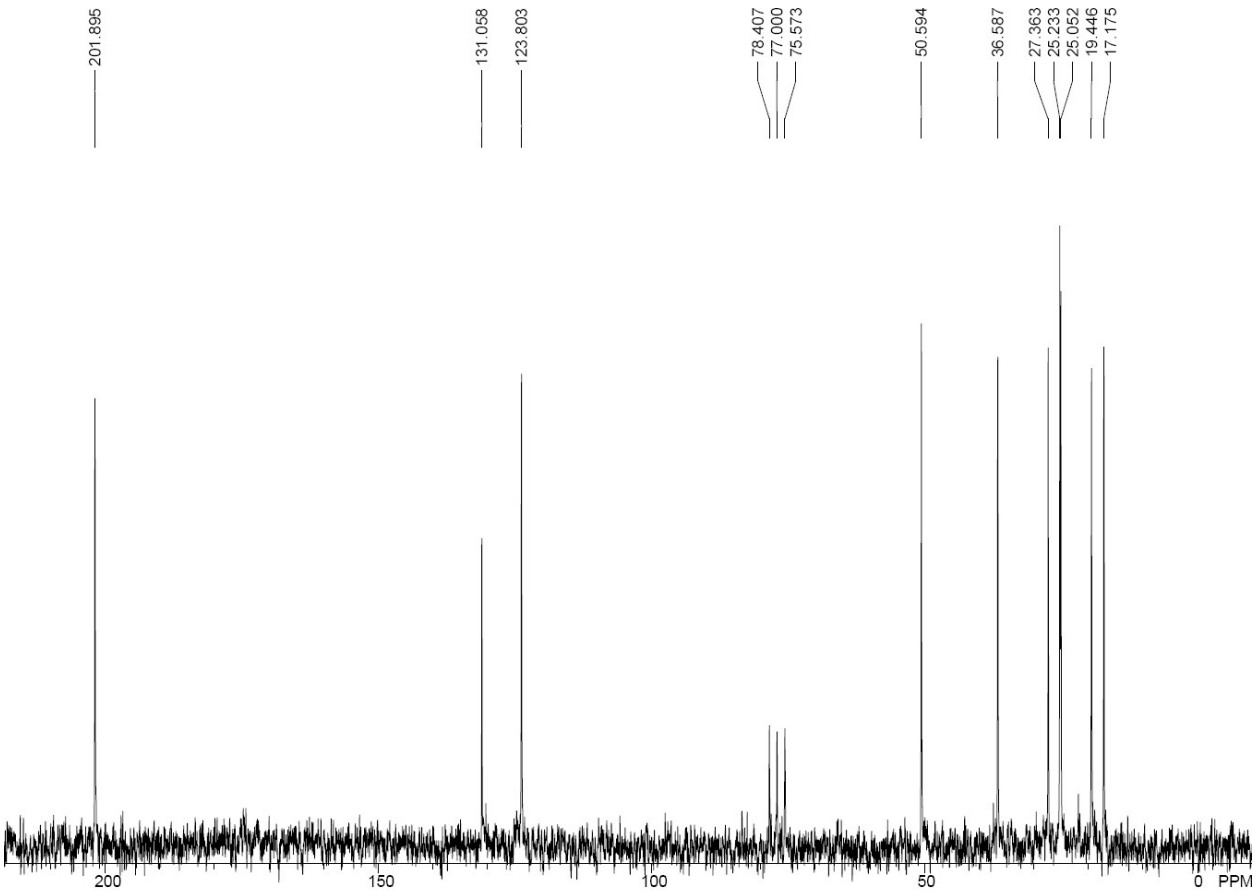
Further Assignments

Further assignments become more difficult. The HETCOR shows that the proton region at 1.8-2.1 correlates to two different carbon atoms, meaning that this region of the spectrum contains overlapped signals.

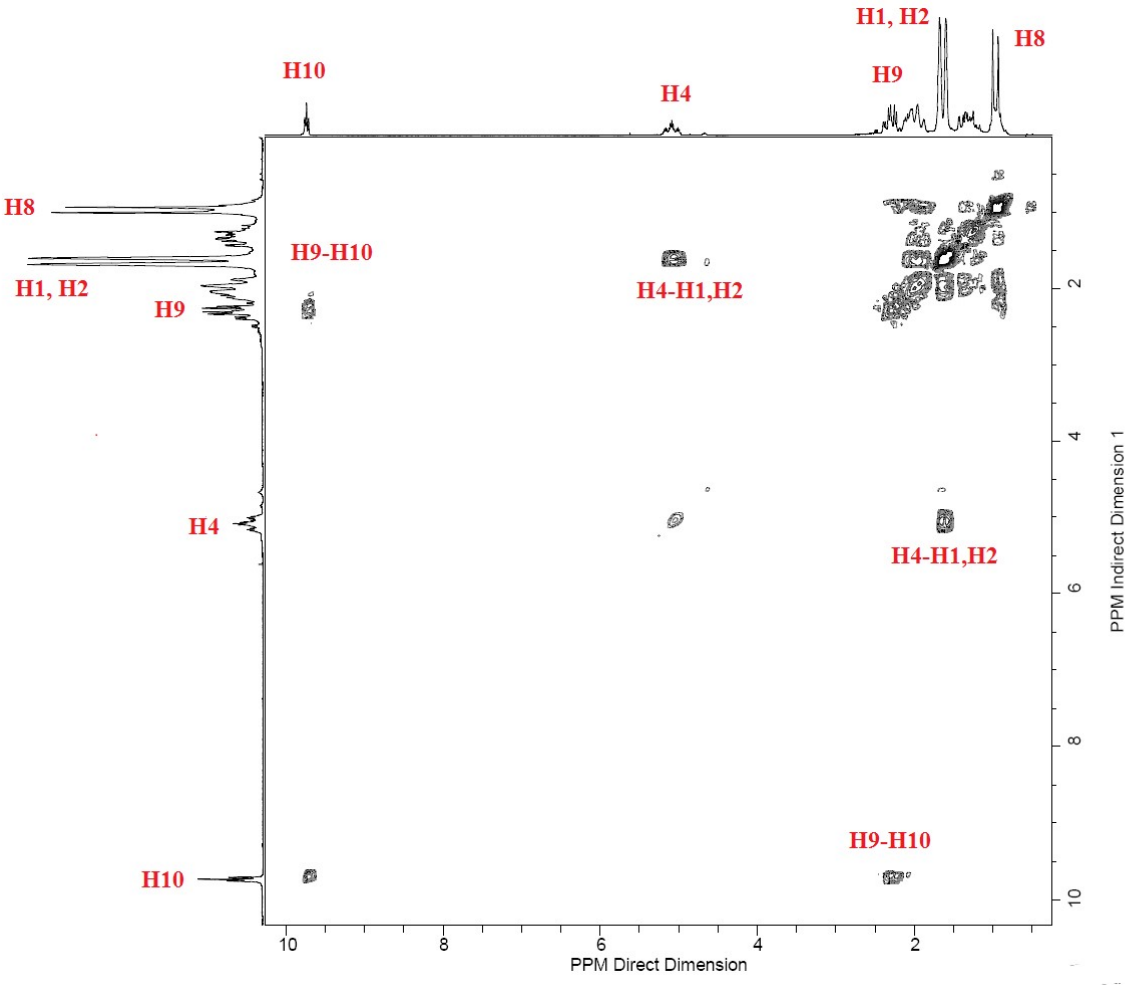
Proton Spectrum



Carbon Spectrum



COSY Spectrum



Heteronuclear Correlation (HETCOR)