6.4: DEPT C-13 NMR Spectroscopy

Learning Objectives

- Understand the difference between 1D ^{13}C NMR and DEPT.
- Determine what information is gained when using DEPT

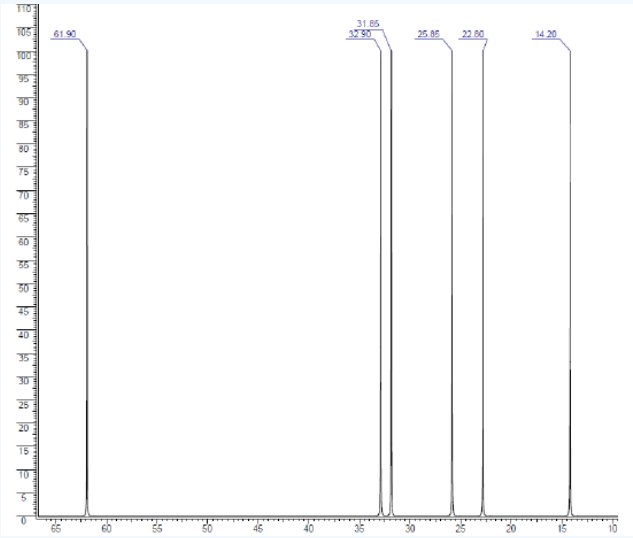
There are many types of experiments that can be run using the NMR spectrometer, including gathering information from ^{13}C NMR spectra and while broadband decoupling gives a simpler spectrum, it loses information about neighbors. Distortionless enhancement by polarization transfer, DEPT, is one of these techniques to gain this type of information back and making it possible to distinguish between methyl (CH_3), methylene (CH_2), methine (CH), and quaternary carbons. In other words, the number of hydrogens attached to a particular carbon can be determined. In DEPT, it takes advantage of the ^{13}C to ^{1}H coupling that is removed in broadband-decoupled ^{13}C spectra.

DEPT experiments often start by running an ordinary ^{13}C spectrum (typically broadband-decoupled spectrum). This allows one to know where the chemical shifts for the carbons in a molecule to be known. For a DEPT, there is a final step in the data acquisition that has a final flip angle of 90 or 135. During the experiment, polarization is transferred from one nuclei to another. Typically, the small gyromagnetic nuclei is observed, so the transfer would be from ^{1}H to ^{13}C. This allows for the DEPT to determine which carbons are attached to hydrogens. In a DEPT-135 spectrum, the CH_3 and CH resonances are upright or positive, the CH_2 resonances are inverted or negative, and quaternary carbons do not show up since these carbons are not directly attached to a hydrogen. Another DEPT experiment is the DEPT-90. While DEPT-135 showed all of the resonances of protonated carbons, DEPT-90 only shows CH peaks (upright/positive).

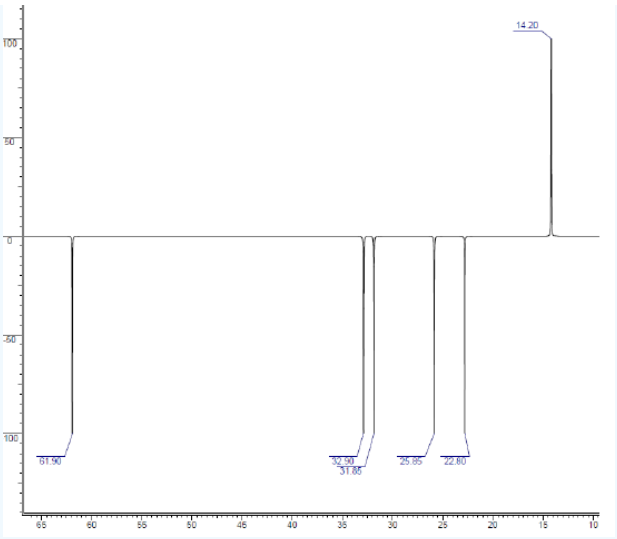
Example 6.4.1

Propose a structure for an alcohol, $C_6H_{14}O$, that has the following ^{13}C NMR spectral data.

Broadband-decoupled ^{13}C NMR spectrum:



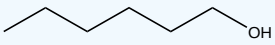
DEPT-135:



DEPT-90: No positive peaks.

Solution

From the ^{13}C NMR spectrum, the resonances inform that there are 6 distinct carbons. There are only 6 carbons in our molecular formula, therefore they are all different. Based on chemical shift, there are no multiple bonds in the molecule. You can also begin by calculating the degrees of unsaturation, which indicates know multiple bonds or rings. The DEPT-135 shows 5 negative peaks and 1 positive peak. The negative resonances are methylenes and the positive peak is either a methyl or methine. The DEPT-90 indicates that there are no CH peaks in the molecule. Therefore, the resonance at 14.20 ppm is a methyl group. Our structure is:

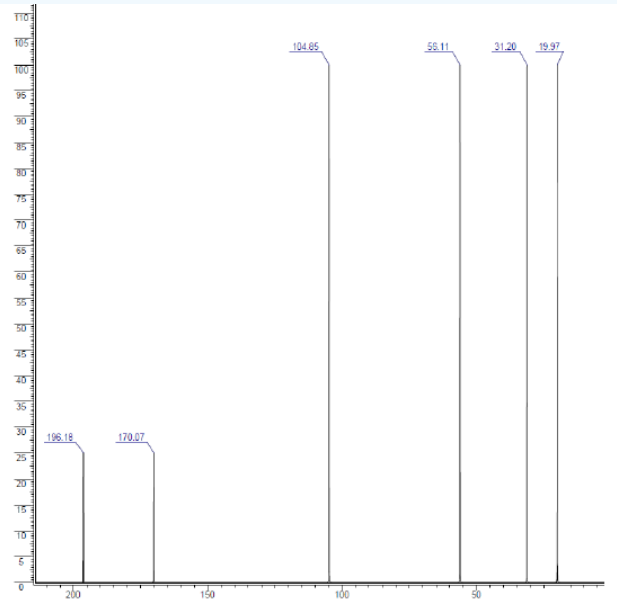


There are many more complicated experiments as well, but that is beyond the scope of this chapter.

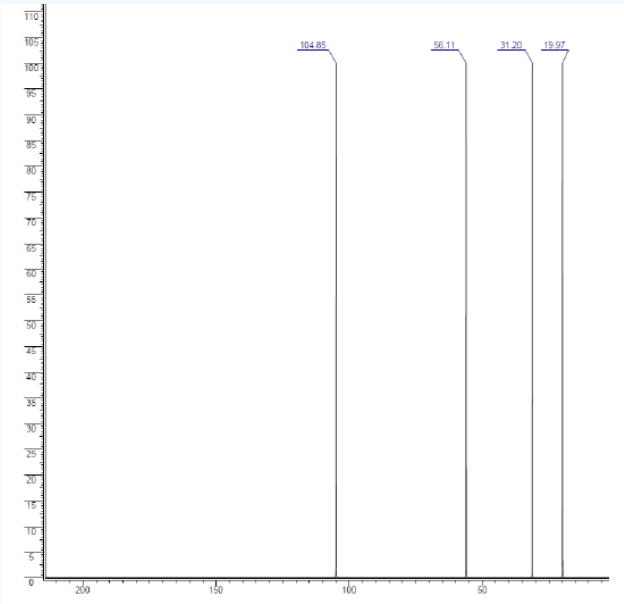
? Exercise 6.4.1

Propose a structure for an alcohol, $C_6H_{10}O_2$, that has the following ^{13}C NMR spectral data.

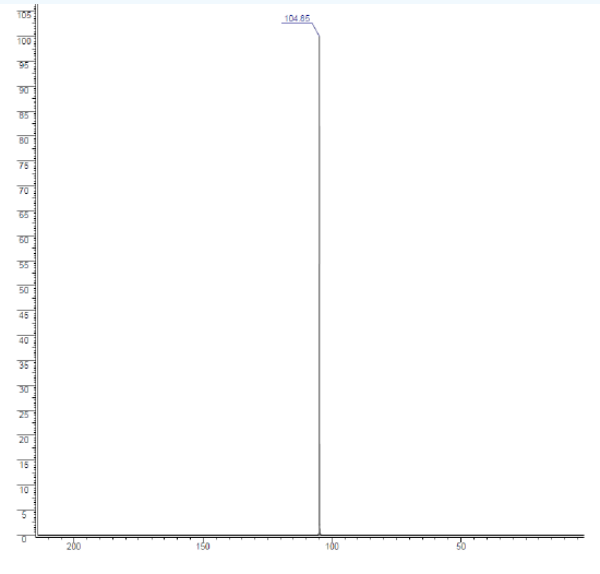
Broadband-decoupled ^{13}C NMR spectrum:



DEPT-135:



DEPT-90:



Answer

There are 2 degrees of unsaturation. The ^{13}C NMR spectrum indicates that all 6 carbons are different. The resonance at 196 ppm indicates a C=O based on chemical shift and the resonances at 170 and 104 ppm indicate a C=C bond. This would account for both degrees of unsaturation. The DEPT-135 shows no negative resonances, so there are no CH_2s. The loss of the resonances at 196 and 170 ppm means that they are quaternary carbons (not attached to hydrogen). The DEPT-90 shows one resonance at 104 ppm, which means it is a CH and before was indicated as part of a double bond. The peaks at 56, 31, and 20 ppm are all methyl groups. The one at 56 ppm must be attached to an electron withdrawing group, which in this example an O. The fragment is $-OCH_3$. To have a quaternary carbon that is not the carbonyl, there must be a methyl group attached to the one of the carbon of the C=C double bond. 196 ppm indicates a ketone, which would be where the last methyl group goes. The final structure based on chemical shift and DEPT is: