13.11: DEPT ^{13}C NMR SPECTROSCOPY

OBJECTIVES

After completing this section, you should be able to

- Understand why the peaks do not have splitting like in ^{1}H NMR.
- Using DEPT, distinguish whether a methyl (CH_3), methylene (CH_2), methine (CH) or quarternary C is present in the molecule and how many.
- Propose a structure based on ^{13}C spectral data.

DEPT (Distortionless Enhancement by Polarization Transfer) ^{13}C NMR Spectroscopy is a powerful technique used in organic chemistry to elucidate the structure of organic molecules. Unlike traditional ^{13}C NMR spectroscopy, which provides only basic information about carbon environments, DEPT enhances signals from specific types of carbon atoms, allowing for more detailed analysis. DEPT works by selectively enhancing signals from three types of carbon atoms: methyl (CH_3), methylene (CH_2), and quaternary carbons (C with no attached hydrogens). This enhancement is achieved through a series of pulse sequences that manipulate the nuclear spins of carbon atoms, resulting in distinct peaks in the NMR spectrum corresponding to each carbon type.

DEPT experiments are used for distinguishing between a CH_3 group (methyl), a CH_2 group (methylene), and a CH group (methine). The proton pulse is set at 45°, 90°, or 135° in the three separate experiments. The different pulses depend on the number of protons attached to a carbon atom. Figure 13.11.1 is an example about DEPT spectrum of n-isobutlybutrate.

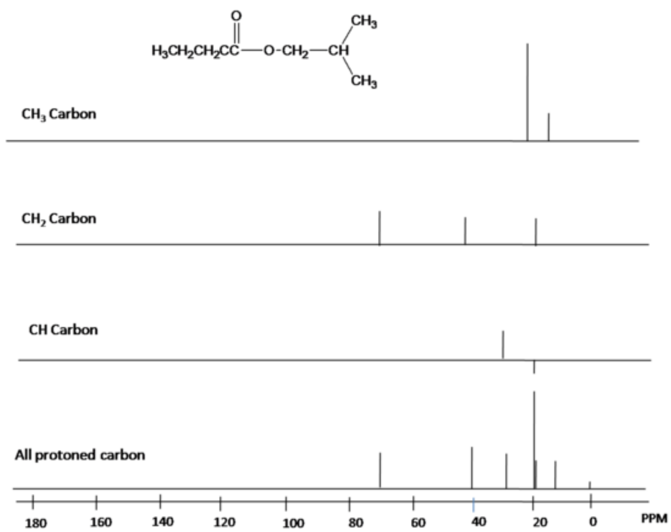


Figure 13.11.1: DEPT spectrum of n-isobutlybutrate.

While broadband decoupling results in a much simpler spectrum, useful information about the presence of neighboring protons is lost. However, another modern NMR technique called DEPT (Distortionless Enhancement by Polarization Transfer) allows us to determine how many hydrogens are bound to each carbon. For example, a DEPT experiment tells us that the signal at 171 ppm in the ethyl acetate spectrum is a quaternary carbon (no hydrogens bound, in this case a carbonyl carbon), that the 61 ppm signal is from a methylene (CH_2) carbon, and that the 21 ppm and 14 ppm signals are both methyl (CH_3) carbons. The details of the DEPT experiment are beyond the scope of this text, but DEPT information will often be provided along with ^{13}C spectral data in examples and problems.

Below are two more examples of ^{13}C NMR spectra of simple organic molecules, along with the type of substitution for that carbon which was obtained from a DEPT experiment.

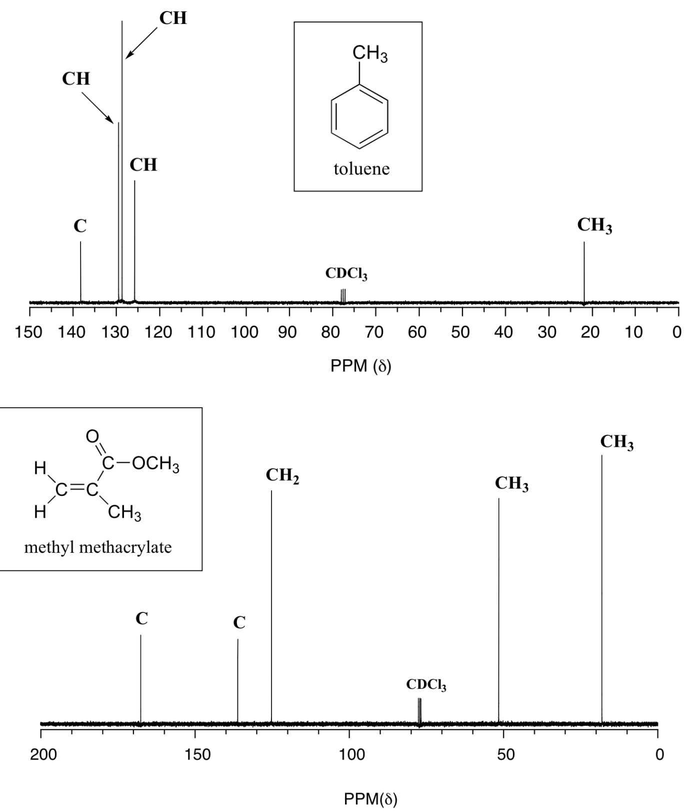
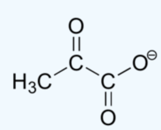


Figure 13.11.2 ^{13}C NMR spectra of toluene (left) and meth methacryalate (right)

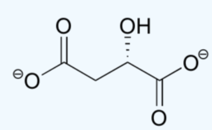
? EXERCISE 13.11.1

^{13}C-NMR (and DEPT) data for some common biomolecules are shown below (data are from the Aldrich Library of ^{1}H and ^{13}C NMR). Match the NMR data to the correct structure, and make complete peak assignments.

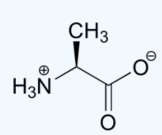
- spectrum a: 168.10 ppm (C), 159.91 ppm (C), 144.05 ppm (CH), 95.79 ppm (CH)
- spectrum b: 207.85 ppm (C), 172.69 ppm (C), 29.29 ppm (CH_3)
- spectrum c: 178.54 ppm (C), 53.25 ppm (CH), 18.95 ppm (CH_3)
- spectrum d: 183.81 ppm (C), 182. 63 ppm (C), 73.06 ppm (CH), 45.35 ppm (CH_2)



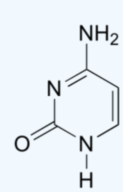
pyruvate



(S)-malate

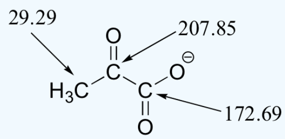


alanine

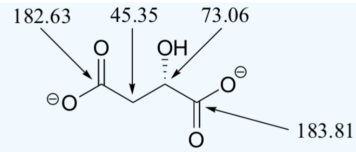


cytosine

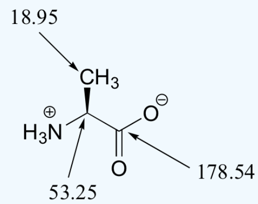
Answer



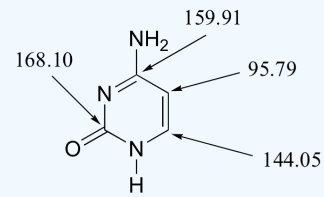
pyruvate: spectrum b



(S)-malate: spectrum d



alanine (spectrum c)



cytosine (spectrum a)