

12.12: ^{13}C NMR SPECTROSCOPY AND DEPT

Distortions Enhancement by Polarization Transfer (DEPT)

DEPT is used for distinguishing between a CH_3 group, a CH_2 group, and a CH group. 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. Fig 11. is an example about DEPT spectrum.

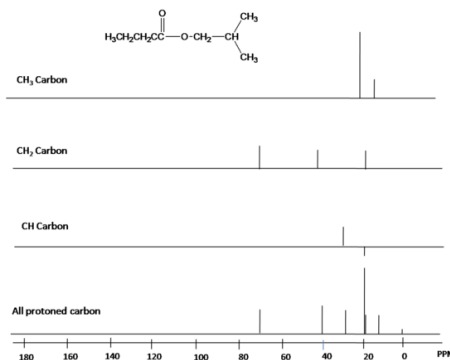
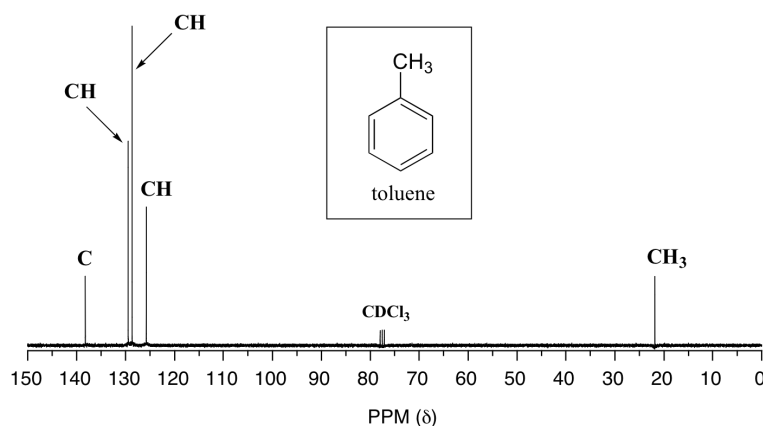
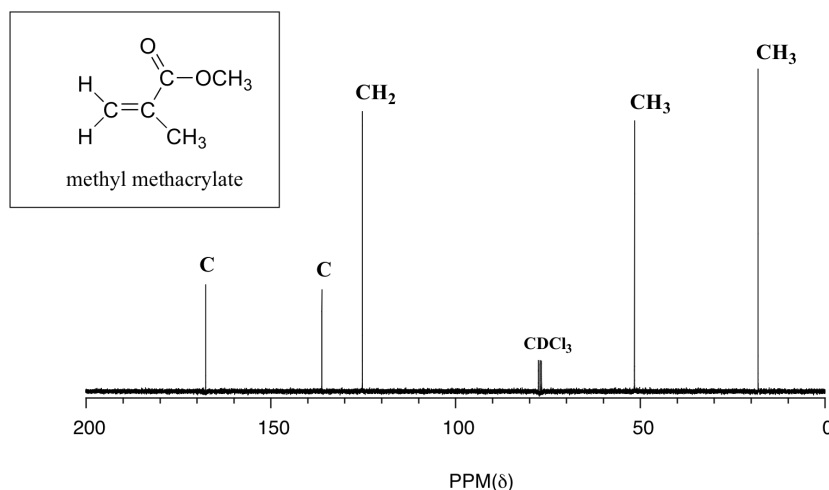


Fig 11. DEPT spectrum of *n*-isobutyrate

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 DEPT information.



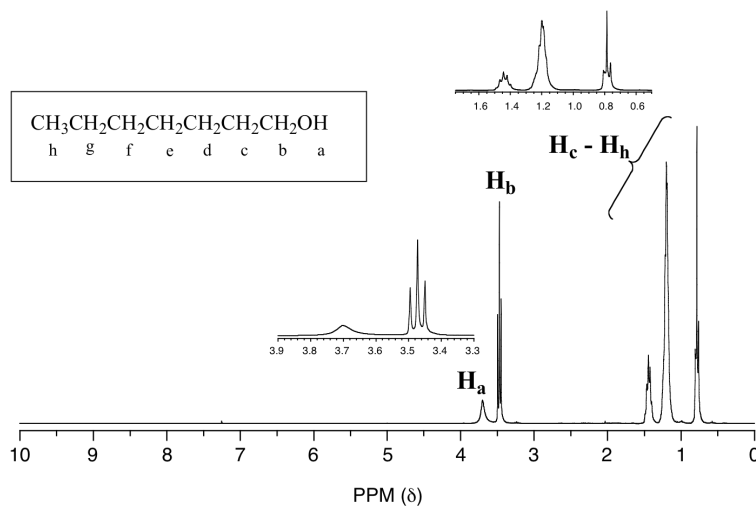


EXAMPLE 13.5.2

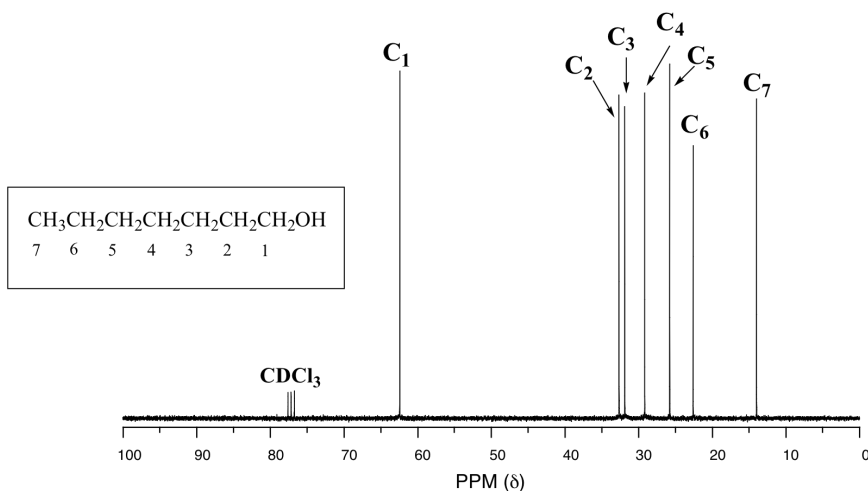
Give peak assignments for the ^{13}C -NMR spectrum of methyl methacrylate, shown above.

Solution

One of the greatest advantages of ^{13}C -NMR compared to ^1H -NMR is the breadth of the spectrum - recall that carbons resonate from 0-220 ppm relative to the TMS standard, as opposed to only 0-12 ppm for protons. Because of this, ^{13}C signals rarely overlap, and we can almost always distinguish separate peaks for each carbon, even in a relatively large compound containing carbons in very similar environments. In the proton spectrum of 1-heptanol, for example, only the signals for the alcohol proton (H_a) and the two protons on the adjacent carbon (H_b) are easily analyzed. The other proton signals overlap, making analysis difficult.



In the ^{13}C spectrum of the same molecule, however, we can easily distinguish each carbon signal, and we know from this data that our sample has seven non-equivalent carbons. (Notice also that, as we would expect, the chemical shifts of the carbons get progressively smaller as they get farther away from the deshielding oxygen.)

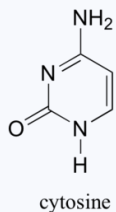
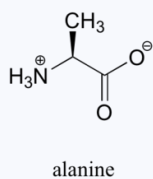
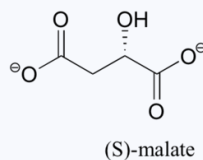
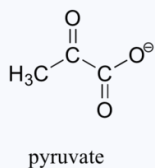


This property of ^{13}C -NMR makes it very helpful in the elucidation of larger, more complex structures.

EXAMPLE 13.5.3

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

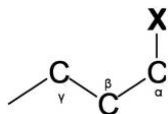
- spectrum a: 168.10 ppm (C), 159.91 ppm (C), 144.05 ppm (CH), 95.79 ppm (CH)
- spectrum b: 207.85 ppm (C), 172.69 ppm (C), 29.29 ppm (CH_3)
- spectrum c: 178.54 ppm (C), 53.25 ppm (CH), 18.95 ppm (CH_3)
- spectrum d: 183.81 ppm (C), 182.63 ppm (C), 73.06 ppm (CH), 45.35 ppm (CH_2)



Solution

^{13}C NMR CHEMICAL SHIFTS

The Carbon NMR is used for determining functional groups using characteristic shift values. ^{13}C chemical shift is affected by electronegative effect and steric effect. If an H atom in an alkane is replaced by substituent X, electronegative atoms (O, N, halogen), α -carbon and β -carbon shift to downfield (left; increase in ppm) while γ -carbon shifts to upfield. The steric effect is observed in acyclic and cyclic systems, which leads to downshifted chemical shifts. Figure 9 shows typical ^{13}C chemical shift regions of the major chemical class.



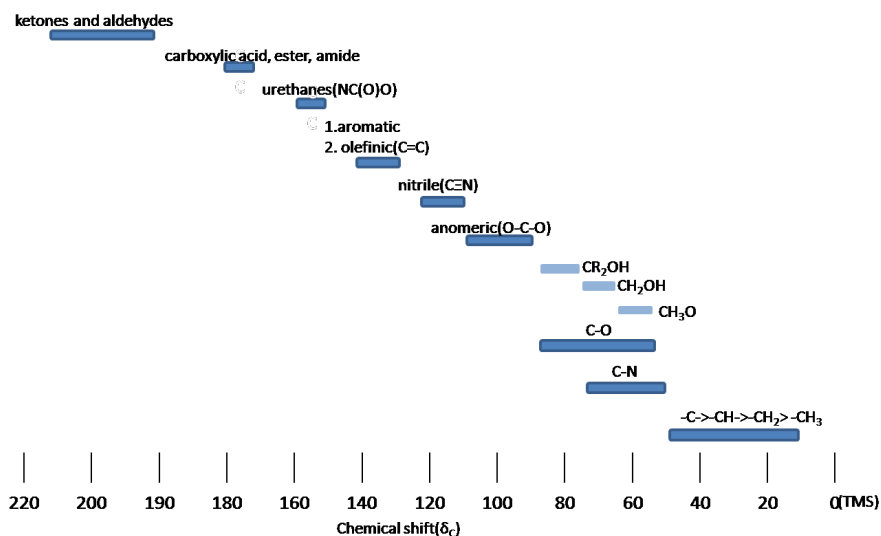


Figure 9: ^{13}C Chemical shift range for organic compound

SPIN-SPIN SPLITTING

Comparing the ^1H NMR, there is a big difference thing in the ^{13}C NMR. The ^{13}C - ^{13}C spin-spin splitting rarely exist between adjacent carbons because ^{13}C is naturally lower abundant (1.1%)

- **^{13}C - ^1H Spin coupling:** ^{13}C - ^1H Spin coupling provides useful information about the number of protons attached a carbon atom. In case of one bond coupling ($^1J_{\text{CH}}$), -CH, -CH₂, and CH₃ have respectively doublet, triplet, quartets for the ^{13}C resonances in the spectrum. However, ^{13}C - ^1H Spin coupling has an disadvantage for ^{13}C spectrum interpretation. ^{13}C - ^1H Spin coupling is hard to analyze and reveal structure due to a forest of overlapping peaks that result from 100% abundance of ^1H .
- **Decoupling:** Decoupling is the process of removing ^{13}C - ^1H coupling interaction to simplify a spectrum and identify which pair of nuclei is involved in the J coupling. The decoupling ^{13}C spectra shows only one peak(singlet) for each unique carbon in the molecule(Fig 10.). Decoupling is performed by irradiating at the frequency of one proton with continuous low-power RF.

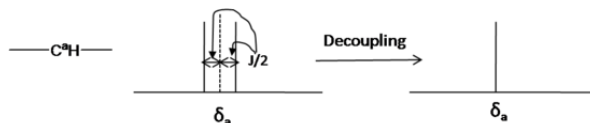


Fig 10. Decoupling in the ^{13}C NMR

CONTRIBUTORS

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