6.6: Uses of C-13 NMR Spectroscopy

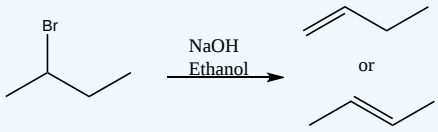
Learning Objectives

- understand how ^{13}C NMR data can be used to distinguish between two (or more) possible structures for an unknown organic compound
- determine if you made the correct product

^{13}C NMR spectroscopy derives information that is helpful for structure determination, especially when paired with ^{1}H NMR spectroscopy. ^{13}C allows the organic chemist a way to determine how many non-equivalent carbons are in a molecule of interest. This allows one to understand if there is symmetry in the molecule or not. The chemical shift of each of the resonances in ^{13}C NMR spectra gives information about the electronic environment, which can indicate what type of functional group is present or what type of bond is present. If you add in DEPT, then the organic chemist can determine how many hdyrogens are attached to each of the carbons.

Example 6.6.1

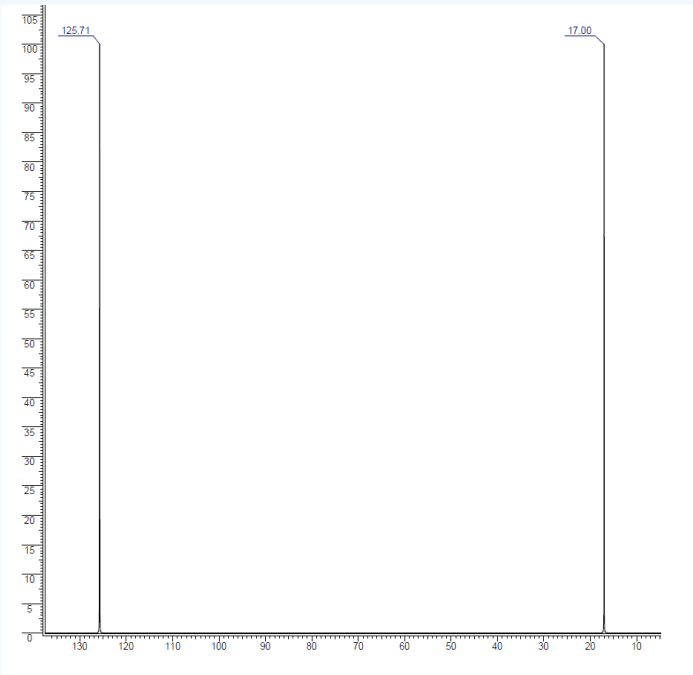
The E2 reaction follows Zaitsev's rule, but how do we know that? Let's consider the reaction below:



1) How can you tell the difference between 1-butene and 2-butene in a ^{13}C NMR spectrum?

2) Which product was made based on the ^{13}C NMR spectrum of the product?

^{13}C NMR spectrum of the product:



Solution

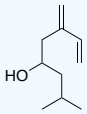
1) IR spectroscopy and mass spectrometry would not be helpful in elucidation of this problem. It is very difficult to address the structural differences in either of those spectroscopic methods. ^{1}H NMR could be used, but there would be overlapping peaks,

so it would again be difficult. In a ^{13}C NMR spectrum, 1-butene would have 4 different signals in the spectrum where as 2-butene would have 2 distinct signals in the spectrum.

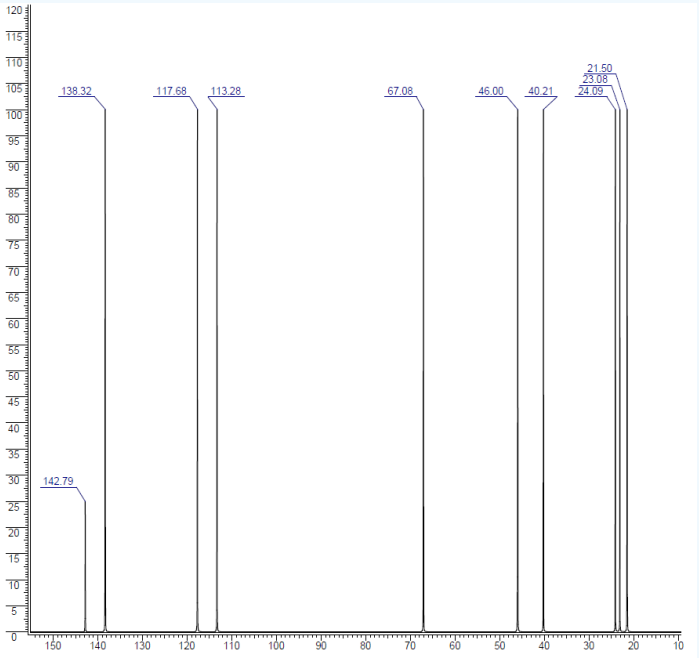
2) With just 2 signals in the spectrum, the product is 2-butene.

Example 6.6.2

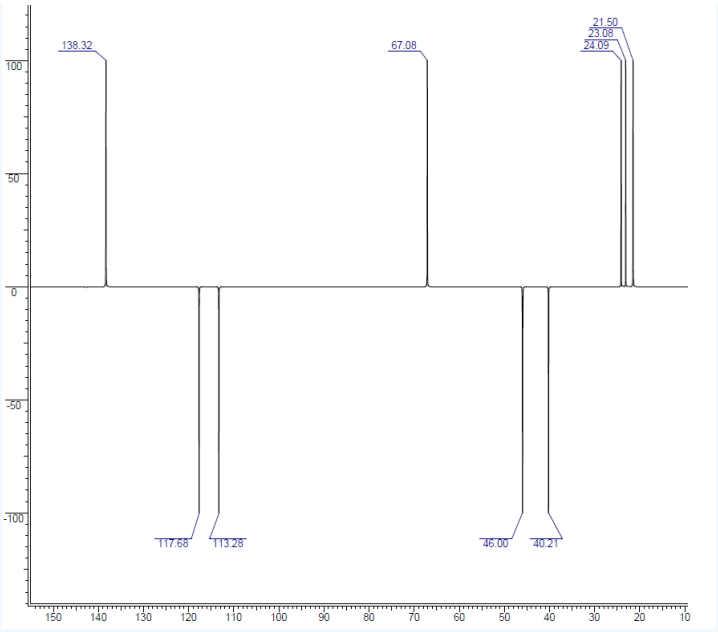
The ^{1}H NMR spectrum had overlapping peaks, which lead to inconclusive results on if the product, ipsenol, was isolated. The structure of ipsenol is below. A ^{13}C NMR spectrum along with a DEPT-135 and DEPT-90 to identify the product.



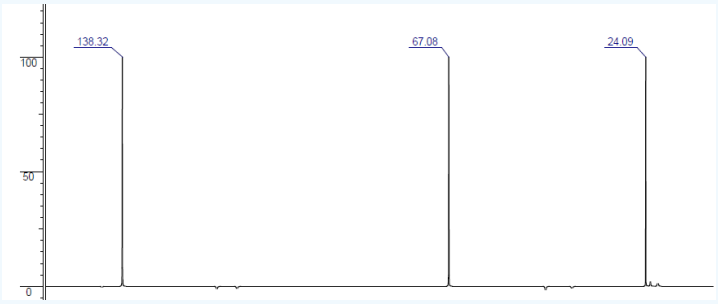
^{13}C NMR spectrum:



DEPT-135 spectrum:



DEPT-90 spectrum:



Solution

Carbon NMR along with DEPT can be a great tool in structure determination. The range for carbon NMR is wider than for 1H NMR, so it can help spread the spectrum out and remove overlapping peaks. In the ^{13}C NMR spectrum, there are 10 distinct peaks, so all of the carbons have been accounted for. If we consider the structure for a moment, then it can be seen that there is 1 quaternary carbon, 3 methines (CH), 4 methylenes (CH_2), and 2 methyl groups (CH_3). In the DEPT-135, the peak at 142.79 ppm has gone away. This is the quaternary carbon, since it is not attached to any hydrogens directly, it is not observed. The negative peaks are methylene groups and there are four - just as expected. There are 5 peaks that are either a methine or methyl group. Finally, the DEPT-90 informs that there are 3 methine groups, which means the peaks at 23.08 ppm and 21.50 ppm would be methyl groups. These 1D-carbon spectra do correlate with the structure.

Contributors and Attributions

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- Organic Chemistry With a Biological Emphasis by Tim Soderberg (University of Minnesota, Morris)