Expt 1 - Identification of an Unknown Compound using IR, MS, EA, Proton and Carbon NMR

Spectroscopy in Organic Chemistry - An Overview

Purpose:
To identify an unknown compound through the use of spectroscopic methods of infrared analysis, elemental analysis, mass spectral analysis and proton nuclear magnetic resonance.

Procedure:
For the next few weeks, we will be examining different methods of analysis of organic molecules. Structural determination will be the primary goal for both the Infrared Spectroscopy and Nuclear Magnetic Spectroscopy. Both methods give different types of information and work well together to help solve the puzzle of unknown structures. Included in this laboratory packet, at this point are several different items that will come in handy along the way. These items include, first, some sample Infrared (IR) spectra of various organic molecules with various functional groups followed by identification problems using IR. This is followed by some samples of mass spectra of different compounds. Then, included are some sample spectra as well as problems for Proton (¹H) Nuclear Magnetic Resonance. As you review and learn the key issues surrounding each of these methods of spectroscopy, you will be issued an unknown sample. You will take an IR spectrum and analyze this for key functional groups. You will be given the elemental analysis (percent composition) for determination of the molecular formula of the molecule. You will be given the mass spectrum for further analysis. Finally, the NMR spectra (proton and carbon) of your sample will need to be analyzed and based on this information you will complete the structural assignment. Your ultimate goal will be the identification of your unknown compound, not from a list but based on your spectral information.

Identification of organic compounds is nothing more than finding the solution to a puzzle. The pieces must all fit together. Typically an organic chemist has the added benefit of knowing what reaction was done, in order to produce the product being studied. Alas, you do not know how any of the unknowns are made. Thus you must rely on physical and spectral data only. When given a liquid or solid, the best place to start would be with a boiling or melting point analysis. While melting points are not too difficult to deal with, boiling point analysis can be an experiment in the level of patience.
a student possesses, or doesn't. If the boiling point or melting point is known, AND the functional group has been identified, a preliminary identification can be made. One would have to view lists and lists of tables of data for molecules possessing the same functional group. Not the easiest way to do this, to be sure.

We then add other information into the puzzle, more pieces so to speak. Information can be gained from physical tests, like solubility tests, where one can identify functional groups based on water-versus-organic solubility, acid-versus-base solubility, etc. This is classically how a functional group was identified before the introduction of IR. Lucky for you, IR is so easy to do now. Solubility tests results can very often be misleading.

Mass spectroscopy is utilized in the 90's as a method of proving a molecular formula, or more exactly, a molecular weight. Before an organic chemist can publish his results of a reaction in a journal, he must prove the structure of the product. To do this, he may either do an elemental analysis (you know - burn the compound in the presence of oxygen and measure the percentages of C, H and O present) or use mass spectroscopy. Mass spectroscopy utilitizes a high-energy electron beam that is passed through a sample. This "excites" the molecule and causes it to shatter. Again, in the past, this was a primary method of structural determination. Organic chemists took the molecular weights of all the pieces and attempted to reform the molecule. The most useful bit of information from the mass spectrum is the "molecular ion", which tells you what the molecular weight of your compound is. We will discuss what information is necessary for you to recognize for the analysis of halides and alcohols.

Finally, we have both carbon ($^{13}$C) and proton ($^1$H) NMR. Both methods of NMR are used by organic chemists at the present time. While the carbon NMR is the simpler of the two, more information is gained from the proton NMR, the background of which was discussed in CHM 331. This session we'll take a look at carbon NMR and see how it can be used to help decipher the structure of an unknown compound.