

Identification of an Unknown Substance using NMR & IR Spectroscopy

You will each be given a sheet with the title "IR/NMR Unknown Spectra." There will be two spectra on it, a ^1H NMR spectrum and an IR spectrum. Each student will be given a different sheet which is identified by a letter written in the "#_____" space at the top of the page. The assignment consists of three parts- identification of the unknown (20 points), one-page report on this identification (12 points) and oral presentation (18 points).

The first thing you should do is to note down this identifier, in case you have problems or you lose the sheet. Then you should work out the molecular formula using the method described on the handout, and check it against the molar mass given at the top. You can send me an email (include the identifier, the molar mass and your proposed formula) and I will confirm that you have the formula correct before you go any further.

With the molecular formula in hand, you can begin to identify your unknown, using the last section of the NMR handout to guide you.

- Calculate the number of "double bond equivalents" (i.e., pi bonds or rings) as described in the handout. Remember that if you have four or more DBEs, your structure probably contains an aromatic ring.
- Work out the number of Hs at each position from the total number of Hs and the total integration height.
- Identify obvious peaks in the NMR spectrum, keeping track of how many carbons, hydrogens, oxygens, etc you have "used up" out of your formula, as well as how many DBEs you have "used up." Follow the method in the NMR handout until you think you have worked out what it is. Note that most of the unknown NMR spectra actually have a list of assignments listing the types of proton and their chemical shifts.
- Identify obvious peaks in the IR, and use this to confirm or eliminate possible structures until you have narrowed it down to one, or at least just two or three. If you cannot narrow it down to one unambiguously, it will be necessary for you to check the literature- I can help you with this.

Report (12 points + 20 points for correct ID):

You should write a short summary of how you deduced the structure of your unknown. This should show your train of logic. When you get close, you should be careful not simply to "guess" a structure and try to "prove" that- rather, you should list all likely structures and eliminate all but the correct one. Be sure to include your identifier, your molar mass and your name, and make sure you don't forget to discuss the IR spectrum.

Oral Presentation (18 points):

You will make a short presentation (7-10 minutes) to your lab section, which will basically be a summary of your report. You need not spend time on explaining how you calculated the molecular formula, or how you calculated the no. of Hs at each position. Instead, concentrate on pointing out the distinctive features of your spectra, and how these led you to your identification of the unknown.