

## *Unknown B*

### Introduction

Unknown B will be the second unknown that you will analyze during this semester. You will utilize the same techniques for unknown B that you did for unknown A.

You will again receive a sample of the compound, a proton NMR spectrum, and an IR spectrum (if solid). Your compound may contain C, H, O, N, or halogens. If it contains N or a halogen, this will be indicated on the bottom of your NMR. If you use up all of your unknown, you can obtain more without penalty, but please try to avoid this where possible.

This unknown will have two different functional groups. Unknowns may contain the following functional groups: alkene, alkyne, alkyl or aryl halide, ether, alcohol, ketone, aldehyde, ester, carboxylic acid, amine, amide, nitrile, and nitro groups.

You are responsible to discover the identity of your unknown without help from your classmates, just as with unknown A. You may ask each other general questions, such as "what page was that IR table on?" but do not ask others for help figuring out what your compound is. If you are stuck or have any questions, come to me for help. I won't confirm whether you have the right compound, but I will let you know if your reasoning is sound, or point out things you may have overlooked. You shouldn't leave the lab until you have figured out what your compound is.

You should keep a record in your notebook of the procedures you perform, the data that you collect, and your reasoning process as you deduce the identity of your unknowns. Make sure to give a brief summary of the procedures you follow when performing chemical tests (enough that you could repeat the procedure with the same materials available). When you have finished all of your lab work and have decided upon a final structure, write a detailed conclusion giving all of the data that supports your decision. Please take a look at the "Sample Lab Notebook for an Unknown" to get a better idea of what your lab report should look like.

### Procedure

#### **Before lab:**

- Review all of the instructions in the lab to make sure you know what you will be doing.
- Write a brief introduction in your lab notebook.
- Review the "Sample Lab Notebook for an Unknown" on the course website.
- Review the "Chemical Tests for Unknowns" on the course website and make sure to bring a copy to lab.
- Review the following techniques on the course website:
  - Characterizing Compounds by IR Spectroscopy
  - Analyzing NMR Spectra
  - Measuring a Boiling Point
  - Taking a Melting Point
- Do the pre-lab questions on-line.

### **During lab:**

Consider the following as you determine the identity of your unknown. It would be best to do the steps below in order, but you can make some changes as needed. For example, if there is a line of people waiting for the IR, start on your boiling point or melting point. If there are no hoods available to take a boiling point but you have taken an IR, you can start on functional group tests. You will want to take a quick look at your NMR when you get started, then come back to it when you are further along.

#### Physical characteristics:

- Look at the sample of the compound that you have been given. Is it liquid or solid? Is it white or colored? Does it have an odor? (Be careful – some of them are pretty strong.) Write it down and make sure you can justify it by the end. (Be aware, though, that some colors, particularly yellow, are a result of impure samples and not an inherent property of the compound. If your compound is slightly yellow but is supposed to be white, you're probably ok. Also, odors are notoriously difficult to describe.)

#### Elemental analysis:

- Elemental analysis can provide information about what elements other than C, H, and O are present. This information is given to you on the bottom of your NMR. If there is a N or halogen, you will see it written there; if not, nothing is written. Once you've looked at this, are there any functional groups you can rule out?

#### Infrared Spectroscopy (IR):

- If you have a liquid compound, take an IR. If you have a solid, one has been provided for you (it's on the back of your NMR). What functional groups are consistent with the bands present? What groups can you easily rule out now? (Don't be too hasty – only rule out things it obviously couldn't be, and make sure you have a good reason.) Refer to the frequency table found in "Characterizing Compounds by IR Spectroscopy" for help with the frequencies of different bands.
- Make sure to label all of the important bands in your compound, and turn your IR in with your report.

#### Nuclear Magnetic Resonance Spectroscopy (NMR):

- Take a look at your NMR. What clues can you learn about the structure? Is the compound aromatic? Are there any tell-tale peaks (aldehydes, acids, vinyl H's, broad peaks indicative of NH or OH)? How many kinds of hydrogen will your structure have? Make sure to label the TMS, solvent, and possible water peaks. I would advise against spending a lot of time trying to deduce the structure at the beginning – it is usually better to wait a few steps and get some confirming data first.
- When you have a good idea of what the functional group is and an accurate melting or boiling point, take a good hard look at your NMR and try to figure out what structures could account for all of the peaks, splitting, and integration shown. Refer to "Analyzing NMR Spectra" for help remembering how to interpret an NMR. Once you have a possible

compound, look it up in the Aldrich catalogue and see if the melting or boiling points match. If so, then you are in business. If not, go back to the drawing board!

- Once you've decided on a compound, make sure to label your NMR spectrum, showing which peaks match up with which H's in the compound. Turn it in with your report.
- If you can't find the name of your potential structure in the Aldrich catalogue, look it up in the formula index near the back of the book. Many of these compounds have common names, and won't be found under the name you'd ordinarily predict. Once you find the correct molecular formula, eliminate any compounds that it can't be (wrong functional group, substituents, etc), and look up any that remain. Use the condensed structures to find the compound that you're looking for. If the Aldrich catalogue doesn't contain the structure you've proposed, it is incorrect – all of the unknowns are found in Aldrich.

#### Melting or Boiling Point:

- Obtain a melting point or boiling point of your compound (depending on whether it is a liquid or a solid).
- Measure boiling points using the instructions in "Measuring a Boiling Point." To save you from chasing wild geese, you may check your boiling points with me to make sure they are reasonably accurate (leave your bp determination set up so that if it's way off you can continue rather than starting over).
- Measure melting points using the instructions in "Taking a Melting Point." Since you don't know the target melting point, you can save time by running two different mp tubes – one quickly to get a general idea, and the second more slowly once you know the target area. If you use only one tube, you will have to run the mp very slowly or you will overshoot the true value. Melting points should be accurate enough that you shouldn't need to check them with me if you have done them correctly.

#### Functional Group Tests:

- Chemical tests are used to confirm the presence of functional groups that you have found in the IR. A list of chemical tests and of procedures for doing them is given in "Chemical Tests for Unknowns." If there is a chemical test for your functional group, you are required to attempt it at least once, even if you are sure you know what it is from the spectra. Please don't use tests to eliminate functional groups which you can already tell by IR or NMR are not there, as this wastes time and material.
- A sample compound containing each type of functional group is available to use as a control so that you can see what a positive test should look like. Unfortunately, chemical tests are sometimes unreliable, and you should give more weight to the spectral data. But they can be helpful, and if nothing else it will help you appreciate how wonderful spectroscopy is :). If a test doesn't give the results you were expecting, use your judgment as to whether you should reconsider your IR data or ignore the test results.

Further information:

- When you think you have determined the structure of your compound, look up its name on the internet. What additional information can you find? Look for toxicity, use, appearance, etc.

Conclusion:

- Your conclusion should include –
  1. your unknown number
  2. the proposed name and structure of your compound
  3. the appearance of the compound and whether it matches the proposed structure (if this information is available)
  4. the melting point or boiling point of your compound compared to the proposed structure
  5. what IR bands the compound has which match your proposed structure
  6. how the NMR peaks fit your proposed structure (be specific!)
  7. whether or not the results of chemical test match your proposed structure

Disposal:

- When you have finished, please dispose of any remaining unknown in the organic waste container. Rinse out the bottle and lid, and put both into the glass waste.