Unknown A

Introduction

During this semester, you will be analyzing three different unknowns. In this lab period, you will determine the identity of unknown A.

With each unknown, you will 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.

Most unknowns have only one functional group, but there are a few that have two. 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. You may ask each other general questions, such as "what page was that IR table on?" but you are not to go around asking others for their ideas on what you might have. If you are stuck, come to me for help.

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. It's ok to write down guesses and false starts. 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, you will write a detailed conclusion giving all of the data that supports your decision.

Before coming to lab, please read all of the instructions, and write out a brief introduction in your lab notebook. Do the on-line pre-lab questions. Then review IR and NMR interpretation, and how to take a boiling point and a melting point. Be sure to bring the pages entitled “Chemical Tests for Unknowns.”

Procedure

1. First, don't freak out. This exercise shouldn't be nearly as stressful as the determination of inorganic unknowns tends to be. I haven't yet had a student who was totally unable to figure out what unknowns they had. The only question is how long it will take and how organized the process will be. If you can start by eliminating the panic factor, so much the better. Please feel free to ask me for help. I won't confirm that you have the right compound, but I will tell you if your reasoning makes sense and help direct you toward any pieces of data that you are ignoring or misinterpreting. Also, don't get overconfident if you think you know what it is. You still have to go through all of the steps!

2. Now, 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 – a few 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.)

3. Look to see if any elements are listed on the bottom of your NMR. Are there any functional groups you can rule out?

4. If you have a liquid compound, take an IR. If you have a solid, one has been provided for you. Look for the bands that we have discussed as obvious indicators. 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

5. Next, look at the 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 locate TMS, solvent, and possible water peaks. I would advise against spending a lot of time trying to deduce the structure at this point – it is better to wait a few steps and get some confirming data first.

6. 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 are accurate enough that you shouldn't need to check them with me.

7. Next, try to pin down the functional group(s) by doing chemical tests. 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.

8. 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!

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

10. Your conclusion should include

- your unknown number
- the proposed name and structure of your compound
- the appearance of the compound and whether it matches the proposed structure (if this information is available)
- the melting point or boiling point of your compound compared to the proposed structure
- what IR bands the compound has which match your proposed structure
- how the NMR peaks fit your proposed structure (be specific!)
- the results of chemical tests which match your proposed structure

Please attach the spectra to the notebook pages you turn in, and label useful IR bands and all NMR peaks on the spectra as well as discussing them in your conclusion!

11. When you have finished, please dispose of any unknown in the organic waste container. Put the bottle and lid in the glass waste.

<u>Questions</u>

1) Why is it important to use a control when performing chemical tests?

2) Why do primary and secondary alcohols give a positive Jones test while tertiary alcohols do not?

3) What compound will result when 2-hexene undergoes the bromine test? What about 2-hexyne? How does this bring about the color change that shows a positive test?

4) What compound will result when 2-hexene undergoes the bayer test? What about 2-hexyne? How does this cause the color of the reagent to change?

5) What should you rule out before using the ferrox test?