Identification of Unknowns

The next two weeks in lab will present you with a challenge--given an unknown organic compound, can you use your knowledge of organic chemistry and spectroscopy to identify its structure? The identification and characterization of new and unknown structures is one of the most important, challenging and rewarding aspects of organic chemistry. Whenever chemists perform new syntheses and transformations, they must unequivocally prove the structure and purity of the compound they have prepared. Usually it is not possible to rely solely on one or two pieces of information to accomplish the characterization; rather, a combination of spectroscopic and chemical data is necessary to prove the structure. Similarly, you will be performing over the next two weeks a series of tests on your unknown compound, in order to prove its structure. Some tests will not be completely conclusive and others will yield easily interpretable results. Some tests may at first appear to contradict one another. You are advised to perform as many tests as are relevant to your compound, so that you have the most complete set of data possible for your determination. **It is extremely important that you make careful observations and keep thorough records in your notebook.** You will be happier if you do not have to repeat your work as a result of carelessness.

Prelab

Prepare your lab notebook by writing a schematic outline of the procedure you will follow over the next few weeks. This should constitute one page. **Read the Identification of Organic Substances document course pack** (pg 427). Familiarize yourself with the Solubility Tests for certain functional groups, the classification tests for alcohols, alkyl halides, alkenes, ketones and aldehydes, and review the chemistry that takes place in these reactions. Read/review "Determination of Boiling Points - Other Methods" on pages 195 – 198 of your Techniques book. We will use a test tube rather than a microscale reaction vessel.

Procedure

Your unknown compound may be an aromatic or aliphatic alcohol, alkene, ketone, aldehyde, ether, halogen, or any combination of these functional groups. Using this information, along with spectroscopic and simple chemical test information, you will identify the exact structure of your compound.

1. Determine the boiling point for your unknown, if it is a liquid. If it is a solid, obtain a melting point.

2. Obtain an IR spectrum of your unknown. Sign up for a time on the blackboard. You may not be able to get it until the second week.

3. While you are waiting to take your IR spectrum, perform solubility tests according to the scheme outlined on p. 433 of the Identification of Organic Substances document in the course pack. *We will not perform the elementary analysis sodium fusion tests.*

4. Perform one or more of the following classification tests in order to identify the functional groups in your molecule. Some tests that we have the chemicals available for you to use are: Chromic acid oxidation (alcohols, aldehydes), iodoform test (methyl alcohols and ketones),

2,4-dinitrophenylhydrazine test (aldehydes and ketones), reactions with bromine (alkenes), Beilstein test (halogens), reaction with silver nitrate (halogens), and the ignition test for aromatic compounds. Make sure that you write the complete equation in your notebook for each reaction performed, and that you understand the meaning and interpretation of a positive result. **It could be very useful to test a reaction on a known molecule (a compound you KNOW is an aldehyde, ketone, etc.), in order to witness a positive result and ensure that your technique is good.**

5. Download an ^{1}H NMR spectrum of your unknown from the NMR instrument, according to the Mestre-C handout. Your instructors will advise you what spectrum corresponds to your unknown.

6. Determine the structure of your unknown. The *CRC Handbook* and *Aldrich Chemical Company Catalog* list boiling points and melting points for compounds. There is also a reference book in the library titled: *The Aldrich Library of FT ^{1}H and ^{13}C NMR Spectra*. It contains NMR spectra for every compound that Aldrich sells. It will be placed on reserve for your convenience. The following website has an extensive database of NMR (^{1}H and ^{13}C) http://www.aist.go.jp/RIODB/SDBS/menu-e.html spectra.

Write-up

After you have determined the structure of your unknown, identify the relevant absorbances in your IR spectrum. Draw a structure of your molecule on your ^{1}H NMR spectrum, label all inequivalent hydrogens (e.g. a, b, c, etc.), and use those labels to assign their resonances in the spectrum. Summarize your data and write a one-page explanation that will *convince* your TA that you know what your compound is. *A statement indicating that your spectra match reference spectra does not constitute an explanation. Instead, explain how the features of your spectra prove your compound's identity and how your chemical test information supports your identification*. Make sure to explain inconsistencies in your data. Do not ignore any piece of information that you acquired.