

Chemistry 212

Exp. 12: Qualitative Analysis

1. The Experiment

This is the final experiment for Organic Chemistry Laboratory. You will be given a small portion of an organic compound, and the objective of the experiment will be to identify it exactly. An extensive list of possible unknowns and derivatives is given in a separate file.

In order to identify your unknown organic compound, you will be allowed to perform 5 types of investigations, in the order listed below:

- a) Visual inspection: Including color, texture, state at room temperature, and flame test;
- b) Solubility tests;
- c) Characterization tests;
- d) Melting-point or boiling-point measurements; and
- e) Synthesis of the derivative, and measurement of its melting point or its boiling point.

* The results of all of the above-mentioned investigations should be recorded in your laboratory notebook as observations and data during the experiment.

- a) The visual inspections will come in handy at the end of the experiment as a check point that could be verified with any reference book used (like Aldrich, Merck index, etc ...).
- b) The solubility tests are the first tests you will perform. They will help you narrow down the classification tests you will do next by suggesting the possible functional group class.
- d) Characterization tests are the old-fashioned way of **determining the functional group/s** present in the unknown. Today, with the advancements made in the field of Organic Chemistry, the spectroscopic methods dramatically facilitate the structure elucidation process. The main objective, however, is for you to become familiar with the characterization tests as well. *Note: IR is NOT allowed in this experiment.*
- e) You will measure the melting point or the boiling point of your unknown in order to **determine the exact identity of the compound.**
- f) To confirm the identity of your unknown a derivative is synthesized. The measurement of the derivatives melting point will enable you to determine the identity of the unknown. *Note: If you have a carboxylic acid, you don't need to make a derivative. Instead, do a titration with a base to find out the molar mass.*

2. The Pre-laboratory Protocol

Read the following sections of the "Microscale Techniques for the Organic Laboratory" book (techniques book):

1. Chapter 10;
2. Pages 43-47 (boiling point determinations).

3. Review the Flame Test you have learned to perform from CHEM 112 lab.

The pre-lab write up for this experiment will be minimal, due to the fact that your decision on which characterization test you need to perform at any point will be dependent upon the outcome of the test before it. Therefore you cannot outline in advance, the series of steps required for identification of your unknown. The only parts that should be pre-written are:

- a) Name, date, title and purpose
- b) A blank table for your unknown # and its physical properties (that you will detect when you obtain the unknown)
- c) A complete solubility flow chart that you will use to indicate your solubility results by *highlighting (required)* the parts of the flow chart relevant to your unknown. The flow chart for solubility can be found in your techniques book, page 271.
- d) Bring the techniques book and the printout of the list of possible unknowns and derivatives to the lab.

3. Procedures and Observations

The remaining procedures, observations, and results sections will be written up as you perform the experiment. The procedures for the derivatives can be found in your techniques book. Be sure to write down all procedures you use in detail.

4. The report

The laboratory report for this experiment will remarkably deviate from the traditional format. The report will be developed in parallel with the progress of the experiment. For this report, I will expect most of it to be typed double-spaced, in Times New Roman 12 or Helvetica 12 font. The following details what I expect and the order that I expect it:

- a) There should be a cover page with your name, the date, and a title that includes your unknown number and identification.
- b) The report body should begin with the purpose, followed by a brief introduction.
- c) Your procedures and observations need not be typed, as they will be attached to the back. Your actual pathway in identifying the unknown must be highlighted on the flow chart.
- d) A discussion of the results should be arranged in the order that you performed the experiments. (See a-e on page one of this handout). A brief outline of the experimental steps taken along with the results and a discussion of those results should be presented. Please use subheadings to visually divide each test. The results of each section should be presented in a table format when there are several pieces of data to report. This should be followed by your interpretation of each result. Finally, express your deductive reasoning for the determination of the exact identity for the unknown. If you were misled, you should explain how you ultimately made your conclusion. The discussion-of-results section will include your logical reasoning, based on the results of all tests performed, which lead you to conclude the identity of the unknown. For each step you should indicate how your choice of compound was narrowed down.
- e) Predict both the $^1\text{H-NMR}$ and $^{13}\text{C NMR}$ spectra.
- f) A conclusion that very briefly summarizes what you have learned at each step and the final choice of identity that you made.
- g) Your copies of this experiment from your lab notebook will be attached to the back of the typed report.

Example order:

III. Results

- a) **Title of tests** (i.e Visual inspection) (The following can be in paragraph form, not necessarily the outline form)
1. *Logical reasoning for doing this test.* (i.e. I made these observations when I received my unknown so that I would know if it decomposed or was contaminated while I was testing it and to narrow down the choices of compound.)
 2. *Outline of procedure* (usually 1-2 sentences.)
 3. *Table of results*
 4. *Interpretation of results* (i.e. My unknown is a solid at room temperature, so it cannot be any of the listed liquids or gases.)
 5. *Conclusion from this test.* (i.e. The visual/ physical tests are inconclusive, but my unknown is a purple crystalline solid so I can eliminate all of the liquids and gases.)