

## Chemistry 222 – Separations and Spectroscopy Spring 2011

This assignment is designed to allow an exploration of some aspects of instrumental analytical chemistry, particularly the areas of spectroscopy and chromatography. The questions are designed to help you understand these topics better and to help support your laboratory endeavors in these areas. A serious effort and one done to avoid a panicked last minute rush to complete the assignment will help you in the long term.

### Goal of Assignment:

The goal of this project is to create a summary of several topics important in common instrumental analytical chemistry, particularly in the areas of spectroscopy and chromatography. Some of these topics will be covered in lecture and in lab discussions. For others, you will be expected to find information to help you using a number of available resources.

### Requirements:

- You may work in groups of up to two (NO MORE THAN TWO) on this project. Working solo is fine, but this is intended as a group effort, accompanied by partners discussing and arguing topics.
- Each person's involvement in the project should be equal! I may try to schedule brief 15 minute discussion in which I ask questions of the group members, expecting equal involvement in the answers to these questions (i.e. equal understanding of the topics). A portion of the grade on this project may be tied to this question session.
- You are to turn in only one set of answers per team containing the names of all group members.
- Report your answers to the questions in an organized and neat fashion. Many of the questions may be best answered by an integration of pictures/figures and words. Handwritten answers and hand-drawn figures are perfectly acceptable, as long as they are legible. Typed answers are also welcomed.
- You must answer the questions IN YOUR OWN WORDS!!!! Explanations direct from the text or web sites are not acceptable and will negatively affect your grade.
- You must use at least one resource from the Internet in each main group of the questions. There are 4 main groups of questions to which this applies. They groups are: molecular spectrophotometry, atomic spectroscopy, chromatography theory, and chromatography techniques.
- No more than 1 typed page of text per answer will be allowed, so be to-the-point. *This does not imply that the answer needs to be 1 page to be correct.* Minimize the "fluff" in an answer and get to the point.
- A bibliography of the resources you used to answer the questions, including Internet addresses used, must be supplied at the end of **each group of questions.**

**Point Value:**

A total of 50 points will be awarded for the spectroscopy section questions and 50 points will also be awarded for the chromatography section questions. These points must be earned by submitting clear, concise, and correct responses to the questions. With a reasonable effort, there is no reason to earn a poor grade.

Scores will be awarded somewhat holistically for each question based on the following scale:

Points Awarded	Evaluation
5	Question answered clearly and correctly, with any numerical results reported to the appropriate number of significant digits and with the correct units.
4	Majority of the question is answered correctly, with slight errors in logic or minor omissions.
3	Reasonable attempt, but minor lapses in logic or understanding, or multiple omissions are evident.
2	A valid attempt was made at the question, but multiple minor lapses in logic or a single substantial error in logic are apparent.
1	A poor attempt was made (something was written down). Little, if any, understanding of the problem was demonstrated
0	No attempt was made at addressing the question.

Failure to include references for your sources will result in a loss of points.

**Possible References:**

A list of the addresses of the internet sites which may provide you some help are posted on the CHEM 222 web site, <http://www2.truman.edu/~blamp/chem222/links.html>. In addition to the Internet, your textbook, lab manual, and Pickler library offerings may also help you in answering the questions.

**Due Date:**

This project will be due by 5 PM on Friday, April 29, 2011. No late papers will be accepted. *A five-point bonus will be given to all papers submitted by the beginning of class on Friday, April 22.* Working on this project in a methodical fashion will aid you in preventing a last-minute rush and should support your understanding of the instrumental portions of Quant. Lab and Lecture. I urge you to make a photocopy of the assignment prior to turning it in, as it will serve as a study guide to help prepare for the Final Examination.

## Questions

### **Spectrophotometry: (UV-VIS Spectrophotometry)**

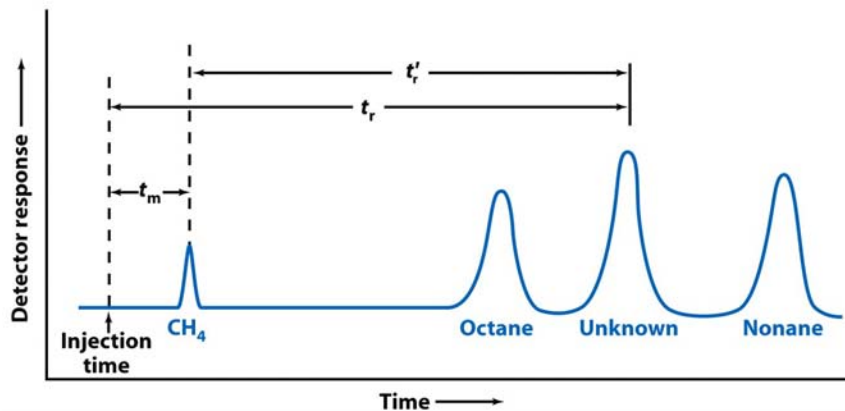
1. When a molecule absorbs light and its color appears blue, what process occurs within the molecule to cause this compound to appear as it does? If the absorbance of this compound were measured over a wide range of wavelengths of light, it would absorb not a single wavelength, but colors of varying energies centering on the wavelength of maximum absorbance. Why?
2. Describe the process by which the transmittance of light through the absorbing sample is measured using a single-beam spectrophotometer, such as the Spectronic-20. Draw a block diagram and identify the key components of a single-beam spectrophotometer.
3. How is the transmittance of light related to the concentration of a light absorbing substance in a way that is convenient and useful to the analytical chemist? (What is the name of the law, the parameters of concern, and how is the determination of the unknown concentration made?)
4. How does a Spectronic-20 spectrometer differ from an array-based instrument like the Ocean Optics USB2000 instruments in use in our CHEM 130 and CHEM 131 courses? Include a block diagram of the key components of the Ocean Optics instrument. Are there any advantages to each type of instrument?
5. What types of molecules typically absorb light in the visible region? What common structural features are often present in such molecules? Give a couple of examples, with structures. (You might want to start by tracking down structures of some colored compounds, like dyes)

### **Atomic Emission Spectrometry: (Flame Atomic Emission Spectrometry (FAES)/Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES))**

1. In an atomic spectrometry experiment, what process results in the emission of light from a gas-phase atom? Why do atoms in the gas phase emit the excess energy in the form of light instead of losing the energy via collisions with surrounding atoms or molecules?
2. The term spectrometry refers to the process of measuring light *emitted* by atoms or molecules in a system. How is this process accomplished in order to generate quantitative data? (Hint: a schematic diagram of a flame emission spectrometer or plasma emission spectrometer will help.) How is this different than absorption spectrophotometry?
3. Describe the process by which a solution-phase sample is introduced into the flame or plasma for a "simple" atomic emission measurement?
4. Increasing the temperature in the flame in an atomic spectrometry experiment can sometimes result in improvement of detection of an element, but can also prove detrimental for the detection of other elements. What causes flame temperature to be this "double edged sword"?

### Separations (Theory):

1. What does a partition coefficient describe in the context of an extraction? If want to extract a solute from 100 mL of solution into another solvent, would it be better to use 100 mL of extractant in one extraction OR to use 100 mL of extractant in ten 10 mL portions? Why?
2. How is chromatography similar to an extraction? Different?
3. Consider the chromatogram illustrated below. For the octane and unknown peaks, calculate the retention time, the adjusted retention time, and retention factor (a.k.a. capacity factor). Also calculate the selectivity factor (a.k.a. relative retention) for the adjacent pair of peaks. Of what significance is each of these terms? What can we learn from them? (You will need to assign a scale to the x-axis in order to perform your calculations.)



4. Efficiency of a chromatographic separation in chromatography is quantified using parameters like plate height and resolution. Qualitatively, what magnitudes are favorable for plate height and resolution? Assuming a 1 meter column, calculate the plate height for the above separation using the octane peak and the resolution of the octane and unknown peaks.

### Chromatography Techniques:

1. Draw a schematic diagram of a simple GC instrument and describe how gas chromatography functions to allow a separation of compounds. Pay particular interest to how a polar compound may be separated from a non-polar compound. What requirements must compounds in a mixture meet for a GC separation to be effective?
2. What differences are experienced when using capillary column GC versus packed column GC? What are the benefits and limitations of each approach?
3. The two most common detectors in GC are a thermal conductivity detector (TCD) and a flame ionization detector (FID). How do these function and what different types of compounds do they detect?
4. Describe how reverse-phase HPLC functions to separate mixtures. In particular, how do the properties of the stationary and mobile phases differ in HPLC compared to GC.
5. Band broadening is a major factor that limits resolution in chromatography. What phenomena contribute to band broadening? How do band broadening considerations differ in HPLC compared to GC?