**Spectroscopy Labs—ICP, uv-vis and IR/Raman/Fluorescence**

*These are one week labs. While there is no official pre-lab assignment, it might be a good idea to look up some information about the expected results for your samples which can help you plan your solution preparation.*

As with chromatography, pairs on a given day can be "A", "B" or "C", and each person in the pair should be either "1" or "2"). Basic schedule below. For specifics of each assignment, read the lab directions. Reports (ICP and uv-vis) are due before you start the next lab. The fluorescence lab questions should be turned in at the end of lab, there is no further report.

| Lab | wk 1 (3/16) | wk 2 (3/23) | wk 3 (4/13) |
|---|---|---|---|
| **ICP (50 pts)** | | | |
| oral report | B1 | C2 | A1 |
| written report | B2 | C1 | A2 |
| **uv-vis (50 pts)** | | | |
| oral report | C1 | A2 | B2 |
| written report | C2 | A1 | B1 |
| **Fluorescence (35 pts)** | | | |
| one list of questions | A1&A2 | B1&B2 | C1&C2 |

**ICP-Inductively Coupled Plasma Optical Emission Spectroscopy**

**Instructions.** Determine the concentration of zinc and of copper in brass. You will be provided with a brass sample when you come to lab. You will need to create your own standards that will work in the 1 – 50 ppm range. I strongly suggest that you use the metals as primary standards and dissolve everything with concentrated nitric acid before diluting with water. Do not use large amounts of nitric acid. You will also need a 1-2 ppm manganese standard in 10% $HNO_3$ to calibrate the instrument. You may use whatever calibration technique you deem appropriate. To save time, you should plan your solution prep before lab. You might also want to google the normal concentrations of zinc and copper in brass so that you know how much to dilute your sample to get into the appropriate range. You need a *minimum* of 25 mL for each solution. Don't forget to make a blank!!! You will want more of the blank, make at least 100 mL of blank.

You will need to turn on the instrument at the beginning of lab, but you will be instructed in its use after you have completed making ALL the solutions.

**Report**

*Written.* Divide your report into 3 sections. The first section should be labeled "INTRODUCTION." In the introduction section include: experimental purpose, justification for using ICP to accomplish this purpose, justification for using the calibration method you chose, brief description of what is brass and how it is used. The second section should be labeled "EXPERIMENTAL SECTION." You should know how to do this from the previous experiments. The rules have not changed. However, you will normally find spectroscopy easier to describe than chromatography so don't worry if it gets shorter. "RESULTS AND DISCUSSION" is the label for the third section. This should contain a clear statement of the percent of copper (and %Zn) in the *original, solid* sample including error. You should also comment on the quality of your results, considering your error, calibration curve and expected values. Include and refer to (perhaps using a phrase like "See Figure 1") your calibration curves. Show your calculations hand-written on the back of the figures. Include a sample calculation for the concentration of ONE standard. Use references throughout the paper as needed, collecting the citations at the end (label that section "REFERENCES" even if you only have one).

*Oral.* Make an appointment at least 24 hours in advance. Bring your lab notebook and calibration curve with calculations written on the back. Be prepared to answer the same questions addressed in the written report. Yes, this does mean it is a good idea to write results of your "literature" search in your notebook, including references.

**UV-Vis Absorbance Spectroscopy**

**Instructions.** Each student in the group should bring a pair of their sunglasses to test. Look up information on sun protection, particularly the health effects of exposure to uv-radiation and the differences for the three uv regions. In lab (in this order): 1) Turn on the instrument (Cary uv-vis spectrometer) to allow sufficient warm-up time; set the maximum absorbance value to 4. Scan ONLY the wavelength region directed in each part of the experiment. 2) Prepare an (about 100 ppm) aqueous quinine solution, measure its uv spectra, determine the value(s) of lambda max. 3) Prepare gaseous toluene by putting a drop at the bottom of the cuvette and capping it. Measure the uv spectrum at 3 different slit widths. (make each slit width significantly different). Focus in on the less energetic peak by narrowing the wavelength region. Observe the differences throughout the spectrum, do not just record $\lambda_{max}$ and absorbance. That is the LEAST significant difference (if it even is different)! 4) Measure both the uv and visible spectra of your sunglasses as well as one of the Myers collection.

**Report**

*Written.* Write a set of step-by-step instructions for general use of the instrument including changing the wavelength regions and slit width. On a separate paper, answer the following questions giving each answer its own paragraph. Write so that the answer is understandable without having to know what the question is. Instructions should be single spaced, answers to questions the usual double space. Attach copies of your spectra. Include directions for getting a print-out.

1) What is the $\lambda_{max}$ for quinine? (Yes, this can be just a sentence. If there is more than one peak/band, report all of them.) Be sure specify the actual solution measured, include concentration and solvent.

2) What is the effect of slit width on the spectra of toluene? Which width is better for qualitative measurements (why)? Which width is better for quantitative measurements (why)? Be specific.

3) Based on your measurements, which sunglasses are best and why? Address both ultraviolet and visible behavior. State specifically what would constitute "ideal behavior" in each region and why that is "ideal" (include references) then describe how closely your sunglasses come to achieving this. Attach spectra so that your descriptions are verified.

*Oral.* Make an appointment at least 24 hours in advance. Bring your lab notebook and spectra (at least sketches if printing it out is not convenient.) Be prepared to answer the questions above as well as describe the experimental conditions (like you did with chromatography). Yes, this does mean it is a good idea to write results of your "literature" search in your notebook, including references.

**Fluorescence**

*Worth 35 pts.* Make two solutions of quinine with significantly different concentrations. One may be the same one you used in the uv (or LC) experiment. Filter some tonic water to remove the bubbles. Obtain a question list from Dr. Myers. Perform the experiments described and answer the questions as you go. Turn in ONE copy of these questions (with answers) before you leave the lab as your report. Make sure both student's names are on this report and both of you are ok with getting the grade associated with this report. Neatness counts, so make sure it is legible. However, there is NO FURTHER REPORT for this experiment.