Syllabus for Chemistry 250 (1 credit)
Instrument Proficiency for Scientists - AA and UV/Vis Spectroscopy
Spring 2008

Instructor

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Office hours 11-12 MWF and 9-10 TR and by appointment

Class schedule

Wet lab work: 2-3 hours per week, specifically scheduled with the instructor to coordinate schedules and avoid conflicts for time on the instruments.
Permissible unscheduled work (not wet lab): computer, literature, writing reports.
Principal locations of course work: GSC 240, 259, 262, 265.
Regular attendance is expected.
Work in groups of two is permitted and encouraged.

Deadline

Submit reports and documentation of completed projects as you finish them (**don't** wait until the end of the term to hand in everything). Completion of all work and submission of reports is Friday, May 9. In place of a final exam, the class will meet at 2:00 pm on Wednesday, May 14, to share experiences and evaluate the course. Come prepared to share/compare the results of your projects.

Course description

This course will enable students to develop proficiency in using two instruments intelligently: Atomic Absorption (AA) and Ultraviolet-Visible (UV-Vis) spectrometers. Students will develop pertinent background through reading, computer based training, and class discussion. Students will develop proficiency in the actual operation of both instruments and in the interpretation of results. Corequisite: Chemistry 242.

Instruments

Buck Scientific Atomic Absorption Spectrophotometer, Model 210 VGP
Ocean Optics PC Plug-In Fiber Optic Spectrometer, Model PC4000 UV-VIS

Supplies

Safety goggles (wear them consistently in lab work)
Permanently bound lab notebook with carbon/carbonless pages. It can be a continuation of a notebook used for another course.

Resources (no texts to purchase)

Primary Resources

Use these resources in place. **Do not remove them!** If you wish, photocopy key sections at your own expense.

1. Instrument manuals for AA and UV-Vis (by the instruments).
2. **ACOL text resources**:
   a. Ed Metcalfe, *Atomic Absorption and Emission Spectroscopy*, Analytical Chemistry by Open Learning, John Wiley, New York, 1987. (QC454 .A8 M48 1987 - Chemistry Seminar Room).
   b. Ronald C. Denny and Roy Sinclair, *Visible and Ultraviolet Spectroscopy*, Analytical Chemistry by Open Learning, John Wiley, New York, 1987. (QD96 .A2 D47 1987 - Chemistry Seminar Room).
3. Computer Based Training on Chem13 (computer by the laser printer in GSC 240).
   a. *UV-Vis Spectroscopy*, JCE Software. **JCE UV-Vis Spec** icon on the desktop. This program requires the original CD, see Dr. Weisshaar. Return it promptly so others can also have access.
   

   b. *Interactive AAS*, Cognitive Solutions Software. **CSS AAS** icon on the desktop.
   

Additional Resources, when appropriate

1. http://www.asdlib.org - Large array of analytical chemistry resources slanted toward the academic lab.
2. Chemistry 311 materials by the window in the Chemistry Seminar Room - lab and writing resources, experimental procedures, articles on instrumental methods, analysis and instrumental methods texts and lab manuals.
3. http://faculty.augie.edu/~dew/ and associated links (DEW's courses website).
4. http://www.bucksci.com/ navigate to atomic absorption spectrophotometers.
5. http://www.oceanoptics.com/homepage.asp navigate to spectrometers.
6. David F. Boltz, Ed., *Colorimetric Determination of Nonmetals*, Interscience, 1958. (QD113 .B6 Mikkelsen Library).
7. Kazunobu Kodama, *Methods of Quantitative Inorganic Analysis: an Encyclopedia of Gravimetric, Titrimetric, and Colorimetric Methods*, Interscience, 1963. (QD101 .K76 1963 Mikkelsen Library).

8. Arnold Weissberger, Ed., *Techniques of Chemistry*, Wiley-Insterscience 1971-. (QD61 .T4 Mikkelsen Library)

Grading

Criteria for grades of C, B, and A are outlined below. Plus/minus grades will be determined by the quality of the work submitted.

To earn a grade of C:

1. Each person maintain a current and satisfactory laboratory notebook. Turn in carbon pages of notebook each week (basket in instructors desk).
2. Be present in lab each week.
3. Study and take notes on the manual for the AA and on the manual and help screens for the UV-Vis instrument.
4. **BEFORE** each lab project, submit a safety report summarizing the hazards, handling, disposal information for the reagents used in each lab project as well as any precautions for using that instrument. Be specific for your lab situation and get the instructor's OK on this information before starting the lab. Be sure to follow your specs.
5. Run the AA method for determination of Zn. Procedure provided as a handout.
6. Run the UV-Vis method for analysis of aspirin. Procedure provided as a handout.

To earn a grade of B - activities for a C plus *one* of the sets below:
To earn an A - activities for a C plus *both* of the sets below:

Set 1

7. Explore the ACOL volumes on AA and on UV-Vis. In a three to four page paper (double spaced), compare and contrast the important aspects of the two instruments and their respective analysis methods. Include in your discussion: a description of the absorption process, differences in absorption band widths for atomic and molecular spectroscopy and the reasons for differences, consequences of the bandwidth on the instrument design, possible interferences for each technique, and any other aspects you feel are appropriate.
8. Work through the **CSS AAS** computer-based training module (GSC 240), documenting your progress in notes and computer printouts.
9. Carry out and document wavelength calibration for the UV-Vis making use of the absorption spectrum of the Ho_2O_3 reference sample. [For future reference: A procedure for calibration of % transmittance or absorbance for UV-Vis instruments can be found in Geraldine Walker Haupt, "An Alkaline Solution of Potassium Chromate as a Transmittance Standard in the Ultraviolet," *J. Research Natl. Bur. Standards*, ***48*** (6), 414-423 (1952).]
10. Generate a standard curve for the **emission** analysis of an alkali metal. Select the metal, identify the appropriate wavelength and concentration range. Write a summary of the procedure used. In a separate section list the necessary modifications to the instrument operations manual. Nyasulu et al. present a procedure that could be adapted in an article in the Journal of Chemical Education http://www.jce.divched.org/Journal/Issues/2007/Mar/jceSubscriber/p456.pdf. Supplemental material for that article is found at http://www.jce.divched.org/Journal/Issues/2007/Mar/jceSubscriber/JCESupp/JCE2007p0456W.pdf.
11. Write a short paper suggesting additions or amendments to the existing instructions for using the AA, including safety and care of the instrument (may be in outline form).

Set 2

12. In a three page paper (double spaced), summarize the interpretation of UV-Vis absorption bands in organic and inorganic compounds, including the more common chromophores. (The project for this set will be obtaining spectra for some of these compounds.
13. Work through the **JCE UV-Vis Spec** computer-based training module (GSC 240), documenting your progress in notes and computer printouts.
14. Write a short paper suggesting additions or amendments to the existing instructions for using the UV-Vis, including safety and care of the instrument (may be in outline form).
15. Obtain absorption spectra for **one** set of samples given below. Select a set and get clearance from the instructor (each group will do a different set to build a library). For each species: adjust the instrument parameters and species concentration (using an appropriate solvent) so the maximum absorbance is 1 or less. For the colorless compounds use the "unsplit" fiber optic cable to get the full UV spectrum. Set the y-axis to display the spectrum full scale. Print the spectrum and save the spectrum file as a processed spectrum.

Sample Set 1	**Sample Set 2**	**Sample Set 3**
aliphatic ketone	acetophenone	nitrobenzene
aliphatic aldehyde	hydroquinone	naphthalene
tartaric acid	benzoquinone	cyclohexene
toluene	aliphatic bromide or iodide	aliphatic amide
$NaNO_3$	$Cu(H_2O)_4^{+2}$ (sulfate)	$Ni(H_2O)_6^{+2}$ (sulfate)
$NaNO_2$	$Cu(NH_3)_4^{+2}$	$Ni(NH_3)_6^{+2}$
$KMnO_4$	$Co(H_2O)_6^{+2}$ (chloride)	$Fe(CN)_6^{-3}$ (potassium)
$MnCl_2$	$CoCl_4^{-4}$	$Fe(CN)_6^{-4}$ (potassium)
rose colored glass blowing goggles	N_2 laser goggles	welders goggles
blue colored glass blowing goggles	Raman goggles	cobalt glass