Syllabus for Chemistry 250 (1 credit)
Instrument Proficiency for Scientists - Raman
Fall 2003

Instructors

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Office hours 8-9 am MWRF and by appointment

Class Schedule

Wet lab work: 2-3 hours per week, specifically scheduled with the instructor, who must be present.
Permissible unscheduled work (not wet lab): computer, literature, writing reports.
Principal locations of course work: GSC 240, 261 (inside 259), and 262.
Regular attendance is expected.
Work in groups of two is permitted and encouraged.

Course Description

This course will enable students to develop proficiency in intelligently using the Raman Spectrometer. Students will develop pertinent background in the theoretical aspects, the operating principles, and interpretation of data through reading, computer based training, and class discussion. Students will develop hands-on proficiency in the actual operation of the Raman instrument and in the interpretation of spectra, coupled with comparison to infrared spectra. (The scheduled time is for the initial meeting; we will quickly move on to hands-on lab time arranged individually or in small groups.) Corequisite: Chemistry 202.

Instruments

Ocean Optics R2000 Raman Spectrometer.
Comparative work with Nicolet FTIR.

Supplies

Safety goggles (wear them consistently in lab work)
Bound lab notebook
Laser safety goggles (provided) are required when the laser source for the Raman spectrometer is in operation.

Resources (no texts to purchase)

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

Primary Resources

- Instrument manuals for Raman (and FTIR as needed). (with the instrument)
- James D. Ingle, Jr. and Stanley R. Crouch *Spectrochemical Analysis*, Prentice Hall, New Jersey: 1988, Chapter 2 pp. 19-22 and Chapter 16 pp. 494-513. (Spectra Collection Shelf, Seminar Room)
- http://nte-serveur.univ-lyon1.fr/nte/spectroscopie/raman/H1TUTO~1.htm Raman Tutorial

Additional Resources, when appropriate

- Kazuo Nakamoto *Infrared and Raman Spectra of Inorganic and Coordination Compounds*, 4th ed., Wiley, New York: 1997. (Spectra Collection Shelf, Seminar Room)
- Gerhard Herzberg *Molecular Spectra and Molecular Structure. II. Infrared and Raman Spectra of Polyatomic Molecules*, Van Nostrand, New York: 1945. (QC451 .H463, Seminar Room)
- Richard A. Nyquist, Curtis L. Putzig, and M. Anne Leugers *The Handbook of Infrared and Raman Spectra of Inorganic Compounds and Organic Salts*, Academic Press, San Diego: 1997. (Spectra Collection Shelf, Seminar Room)
- Dennis P. Strommen and Kazuo Nakamoto *Laboratory Raman Spectroscopy*, Wiley, New York: 1984. (Spectra Collection Shelf, Seminar Room)
- Chem 311 collection by the window in the Seminar Room.

Computer Resources
http://www.oceanoptics.com/homepage.asp
http://www.oceanoptics.com/products/raman.asp
http://www-lasir.univ-lille1.fr/newlinks.htm
http://faculty.augie.edu/~dew (home page for Dr. Weisshaar's courses)
http://ed.augie.edu/~viste/Raman/OperatingInstr.pdf
http://ed.augie.edu/~viste/Raman/RamanSoftware.html
http://ed.augie.edu/~viste/Raman/Raman2JdxHelp.html
http://ed.augie.edu/~viste/Raman/JdxViewHelp.html
http://www.jcamp.org/
http://www.jcamp.org/protocols.html#ir4.24
http://www.jcamp.org/protocols/dxir01.pdf

Deadline
Completion of all work and submission of reports is Friday, December 5. In place of a final exam, the class will meet at 2:00 pm on Wednesday, December 10, to share experiences and evaluate the course.

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. Maintain a current and satisfactory laboratory notebook, individually.
2. Be present in lab each week.
3. Study and take notes on the manual and instructions for the Raman.
4. Prepare samples for Raman spectra: liquid (filter and out-gas) and solid samples (extra in-line laser filter).
5. Wearing laser safety goggles, record Raman spectra of small organic molecules belonging to these families: alkane, alkene, alkyne, aromatic, alcohol, aldehyde, ketone, carboxylic acid, ester, nitrile. At least two of these should be solids. Create a folder for your group in C:\Students and save the spectra in *.ram format in that folder.
6. Print each spectrum from #5 and label (hand written is OK) the band(s) characteristic of the primary functional group in that family (for example: for the ketone, identify the band(s) arising from the carbonyl group).
7. In a written report, summarize safety precautions and proper care of the Raman spectrometer. Include laser safety, fiber optic probe care, disposal information for the compounds used in #5. Clear your disposal plan with the instructor BEFORE you dispose of any samples.
8. Successfully demonstrate to the instructor your operation of the Raman spectrometer.
9. Clean up all your samples following your disposal plan. Clean up the sample bottles and syringe filter adapters you used and return them to the appropriate boxes by the Raman.

To earn a grade of B - activities for a C plus:
10. Explore the selected sections of Ingle and Crouch and the Raman tutorial at nte-serveur.univ-lyon1.fr/nte/spectroscopie/raman/H1TUTO~1.htm and summarize the most significant aspects in a 4 page paper (double spaced). Include in your discussion: Theoretical origin of the Raman spectrum, comparison of Raman and IR spectra, instrument design considerations (especially those related to our instrument), wavelength and intensity calibration.
11. Use the Nicolet FTIR to obtain the FTIR spectra for three of the compounds from #5. Consult with the instructor if you are not certified on the FTIR. Create a group folder in C:\Students and save the spectra there.
12. For the compounds in #11, export the Raman and the FTIR spectra as text files (Raman - File/Export Spectra, FTIR - SaveAs .csv file). Consulting the handout, import these into a spreadsheet and plot Raman and FTIR spectra on the same axes (one graph for each compound). Correlate observed bands in FTIR and Raman spectra with all the functional groups in each compound. To the extent possible identify the vibration responsible for each band (e.g. C-H symmetric or asymmetric stretch). Which bands/vibration modes are active in both Raman and FTIR? Which are active only in IR? Which are active only in Raman? In a 4-5 page paper (double spaced), summarize the interpretation of the Raman and FTIR spectra for these three compounds.

13. Raman spectra inorganic compounds:
    a. Select three inorganic compounds that are soluble in water; include at least one oxoanion and one transition metal (not necessarily in the same compound).
    b. Prepare a safety report summarizing the potential hazards, handling precautions, and a disposal plan for each compound. Clear this with the instructor before making or disposing of any solutions.
    c. Make aqueous solutions of sufficient concentration to produce good spectra.
    d. Obtain spectra for each of your solutions. Save the spectra in your folder and print out each spectrum.
14. Clean up all your samples following your disposal plan. Clean up the sample bottles, syringe filter adapters and FTIR cells you used and return them to their appropriate locations.

To earn a grade of A - activities for a B plus:

15. Interpret the Raman spectra of your inorganic samples (correlate bands with functional groups and vibration modes). Document your interpretation on the printed spectra, adding a page of further explanation if necessary.
16. Quantitative Raman:
    a. Choose one of the organic or inorganic compounds you explored previously that is soluble in water. Prepare at least 5 aqueous solutions varying the concentration enough to significantly change the intensity of the spectral peaks, with the absorbance of the highest concentration around 1. Acquire and save spectra for each solution and for pure water. Save your solutions until the instructor has approved your results (see below).
    b. Provide the instructor with your most concentrated solution (enough to dilute for an unknown) and the range of concentrations you studied. He will prepare an unknown solution that falls within that concentration range. Obtain the spectrum for that solution. Save the solution.
    c. Choose an intense absorption band that does not overlap with a band in your pure water spectrum and measure the absorbance at peak maximum for each of your solutions and your unknown. The peak maximum should occur at the same wavelength in each spectrum.
    d. Using a spreadsheet, prepare a plot of Absorbance vs. Concentration (calibration curve) and determine the equation that best fits the line (regression line). Beer's law (A=abc) predicts that the calibration curve should be linear. Is this the case for your solutions? Consult a Chem 120 or 242 lab manual for a refresher on using a spreadsheet to analyze data.
    e. Use the regression equation to calculate the concentration in your unknown solution.
    f. Turn in copies of the spectra for your solutions, a printout of the spreadsheet including the calibration curve showing your data as markers, the regression line as a line only, and the regression equation. Check the accuracy of your results with the instructor. If your accuracy is not acceptable, you will have to rerun the experiment (that's why you saved your solutions).
17. Clean up all your samples following your disposal plan. Clean up the sample bottles and syringe filter adapters you used and return them to the appropriate boxes by the Raman.