

CHEMISTRY 426
INSTRUMENTAL ANALYSIS
WINTER QUARTER 2008

Instructor Norman J. Dovichi Office Hours: MT 1:30 – 2:20 PM
Chem Library, Room 131 dovichi@chem.washington.edu

Lecture MW/F 9:30 - 10:20 AM, Bagley 261

Text "Principles of Instrumental Analysis," 6th Edition,
by Skoog, Holler, and Crouch

Webpage <http://faculty.washington.edu/dovichi/CoursePages/chem426.html>

Lab Manual TBA

Lab Czar Tom Leach, BAG 133D

Lab and Teaching Assistants

| Section | Day | Time | TA. |
|---------|-----|----------------|------------|
| AA | T | 1:30 - 4:20 PM | Roza Wojik |
| AB | W | 1:30 - 4:20 PM | Yehan Li |
| AC | Th | 1:30 - 4:20 PM | Roza Wojik |
| AD | F | 1:30 - 4:20 PM | Yehan Li |

Course Objectives

- (1) Understand the principles of operation of the major classes of modern chemical instrumentation.
- (2) Use modern chemical instrumentation in a practical setting to analyze real samples.
- (3) Evaluate the performance of an instrument in terms of figures of merit such as limit of detection, linear dynamic range, and resolution.
- (4) Compare various measurement approaches for applicability to specific analytical situations.
- (5) Use a high-level numerical analysis environment, MATLAB, to analyze data.

Course Content

The lectures will cover three classes of instrumentation: Spectroscopy, Chromatography, and Flow Injection Analysis.

The experiments have been designed to demonstrate the use of modern chemical instrumentation in the analytical laboratory. All experiments will involve "real world" analytical problems and unknowns. The first laboratory will acquaint the student with the high-level numerical analysis software, MATLAB. MATLAB will be used for data analysis in almost all experiments. Following this there will be 8 sessions with various chemical instruments organized as two groups of 4 experiments. Students will work in pairs for all experiments except the first MATLAB experiment, rotating among the experiments, shown below.

Note that you will need to provide your own 3 1/2 inch diskettes for computer work.

| | |
|----------------------|-------------------------------------|
| Grading | Expect a class average of about 3.2 |
| Exams | 60% (30% for each of two exams) |
| Lab Work and Reports | 40% |

No makeup exams will be given. If you have an unavoidable excuse for the first exam, the weight of the exam will be transferred to the final.

A short report is assigned for all labs. Lab reports are due at the start of lab on your next lab period, with the exception of the MATLAB experiment, which is due two weeks after your first lab period. Your final lab report is due one week after your last lab. You must turn your lab report on time to receive full credit; 10% of the total points will be deducted for each day that the report is late.

Lab work must be started on time. Arriving at your lab more than 15 minutes late will not be tolerated. After the first infraction, late students will not be allowed to work, with no make-up lab allowed, resulting in a zero for that lab.

Upon completing a lab and before leaving the lab area, you must have your T.A. initial the page(s) of data you will eventually be handing in with your lab report.

LECTURE SCHEDULE

| Date | Topic | Chapter |
|-----------|---|---------------|
| Jan 7 | M Introduction, MatLab, statistics | 1, 5 |
| 9 | W Data Analysis, signal and noise | 5, appendix 1 |
| 14 | M Lab Rotation, Exp. 2-4 | |
| 16 | W Data analysis, calibration | 5 |
| 21 | M Martin Luther King Day, NO CLASS | |
| 23 | W UV-Vis absorbance principles | 6, 7, 13 |
| 25 | F UV-Vis absorbance principles | 6, 7, 13 |
| 28 | M Absorbance instruments | 13, 14 |
| Feb 4 | M Fluorescence | 15 |
| 6 | W FT-IR | 16, 17 |
| 11 | M Elemental spectrometry | 8, 9 |
| 13 | W EXAM ONE | |
| 18 | M Presidents' Day, NO CLASS | |
| 20 | W Mass spectrometry | 20 |
| 25 | M Mass spectrometry, surface analysis | 21 |
| 27 | W GC and GC-MS | 26, 27 |
| March 5 | W LC | 28 |
| 7 | F Electrophoresis | 30 |
| 12 | M Automated methods of analysis | 33 |
| 14 | F Review | |
| 19 | W FINAL (8:30-10:20 AM Bag 261) | |

Note: three lectures will be given on a Friday (Jan 25, Feb 18, and March 14).

Description of Laboratory Experiments

Group 1 January 15 – January 18

1. MATLAB Learn MATLAB as data analysis tool

 Choose Lab Partner

 BAG 48
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Group 2: January 22 – February 8

2. FT-IR Vibrational Spectroscopy, Regression Analysis
 BAG 83
 3. UV-Vis Absorption Electronic Spectrophotometry, Multicomponent Analysis
 BAG 83
 4. HPLC High Performance Liquid Chromatography
 BAG 83
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Group 3: February 12 – March 4

5. Capillary Gas Chromatography
 - Mass Spectrometry Separation of a Mixture, Library Searching
 BAG 83
 6. Fluorescence Excitation/emission & plate reader
 BAG 83
 7. ICP-MS or -emission Metals Analysis
 BAG 133A through BAG 191
 8. Flow Injection Metals Analysis, Automated Sample Preparation
 Analysis BAG 83
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Lab Work**General Information**

1. You will not be assigned a desk for this laboratory. Lab work is done at various locations, as listed above. Computers are available for data analysis and report writing.
2. Most of you will be working in pairs (labeled A, B, C and D in Laboratory Schedule). It is desirable that each partner obtains her/his own data. In rare cases, this will be impossible. *However, each person must submit a separate, independent lab report.* Copying or paraphrasing of a partner's report will result in a mark of zero on that lab for both partners.
3. Before leaving the laboratory, you must submit your data to the TA or the instructor for preliminary evaluation and initialization. Make sure that you have properly restored the instrument to its stand-by condition.
4. Please come to the laboratory prepared. Read the manual carefully before performing the experiments. The TA will be happy to answer any questions at the beginning of the lab period. Note that there are references at the beginning of some of the experiments. These are on reserve in the chemistry library, Chem 426 Answer Key. Please read them before embarking on the experiments.

LABORATORY SCHEDULE

| Week/Pair | A | B | C | D |
|-------------|---------------------------------------|---------|---------|---------|
| 1/15 - 1/18 | MATLAB Experiment | | | |
| 1/22 - 1/25 | Expt. 4 | Expt. 3 | Expt. 2 | Expt. 2 |
| 1/29 - 2/1 | Expt. 2 | Expt. 2 | Expt. 4 | Expt. 3 |
| 2/5 - 2/8 | Expt. 3 | Expt. 4 | Expt. 3 | Expt. 4 |
| 2/12 - 2/15 | Expt. 5 | Expt. 6 | Expt. 7 | Expt. 8 |
| 2/19 - 2/22 | Expt. 6 | Expt. 7 | Expt. 8 | Expt. 5 |
| 2/26 - 2/29 | Expt. 7 | Expt. 8 | Expt. 5 | Expt. 6 |
| 3/4 - 3/7 | Expt. 8 | Expt. 5 | Expt. 6 | Expt. 7 |
| 3/11 - 3/14 | OPEN, reserved for special situations | | | |
