

## CHEM 5650 - INSTRUMENTAL ANALYSIS LABORATORY

Spring 2006

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Office Hours: MWF from 1:30–2:30 PM

Laboratory Meetings: Tuesday/Thursday 8:30–11:30; ML 362 and ML 364

Text: Principles of Instrumental Analysis, Skoog, Holler and Nieman, 5 th Ed.

Laboratory Procedures: Individual handouts provided for each of the laboratories

T.A.: Prakash Joshi email: [prjoshi@cc.usu.edu](mailto:prjoshi@cc.usu.edu)

### Laboratory Grading:

Specific Lab Reports (8 @ 100 pts/report)	800 pts
Special Project Design & Formal Report	200 pts
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<b>Total Points</b>	<b>1000 pts</b>

Final laboratory grades will be assigned based on University policy using the following minimum percentages; A: 90-100%, B: 80-89%, C: 70-79%, D: 60-69%, F: below 60%. Plus and minus grade modifications are assigned according to University policy. Note: Grade cutoffs may change to lower percentages (but not higher) depending upon the overall performance of students in the laboratory.

**Objectives:** Instrumental analytical chemistry is a constantly evolving discipline. Analytical chemists constantly strive to improve the sensitivity, speed, and accuracy of established analytical techniques, to extend existing techniques to new analytical problems or applications, and to invent new instrumental tools for chemical and biochemical analyses. This laboratory is designed to supplement the Chem 5640 classroom instruction by familiarizing students with established instrumental techniques currently used in industrial and academic analytical laboratories. The overall goal of this laboratory is for the student to learn how to use analytical instrumentation to solve chemical problems. Analytical problem solving is a three-step process. First, you must recognize the nature and scope of the problem to be solved and identify those chemical and physical properties of a sample that can be exploited for providing the necessary information to help solve the problem. Second, you must learn to assess the relative merits of competing techniques and select an instrumental method that is most appropriate for the problem. Last, you must learn how to interpret your results within the limitations of the instrumental method used to obtain them. These problem-solving skills are sufficiently general that you should be able to apply them to new chemical analysis problems later in your career.

**Laboratory Safety:** Safety regulations are to be observed at all times. Failure to obey safety rules during a laboratory will result in a point penalty awarded for that laboratory. Repeated offenses will result in dismissal from the laboratory. Laboratory points will also be deducted for failure to clean up and properly dispose of used chemicals at the end of the laboratory period.

**Notebooks:** All students must maintain a bound laboratory notebook to record and organize experimental results. Appropriate notebooks are sold through the University bookstore. This notebook must have duplicate (carbon) pages which will be removed and turned in to your instructor after every laboratory period in which data is collected. Notebooks should be organized as follows:

**Table of Contents:**

Leave a couple of pages for a Table of Contents; keep it up to date during the semester. Data in your notebook should be entered on consecutively numbered pages. Date every page that data is taken on. **Start a new page for each day's experiment.**

**For each lab experiment, include the following information prior to the start of the laboratory period (instructor or TA may check during the laboratory and take off points for the lab if not completed):**

Title of the experiment

Date experiment was conducted

Objective: A brief statement of the experimental goals.

**During the laboratory make sure to record:**

Laboratory Coworkers for that day's laboratory

Procedural Notes: Include any modifications to the general procedure provided or any unusual observations or problems encountered.

Tabulated Data: Include all data collected as part of the experimental procedure.

Qualitative data collected (like spectra) should be printed out and added to the student's notebook (leave a space to tape them in, you may cut and paste them, reducing them in size if necessary to fit) after the class period.

Calculations: Include preliminary calculations and any calculations performed during lab.

**Laboratory Fees**

A laboratory fee is required for this course. The laboratory fee is used to pay for reagents and to help maintain the instrumentation and glassware.

## List of Chem 5650 Instrumental Analysis Laboratories for Spring 2001

3 Students per lab group (There will be two, four week rotations of experiments)

- Laboratory A**    Electronics Laboratory
- Laboratory B**    Atomic Absorption Laboratory
- Laboratory C**    Ion Selective Electrode/pH electrode Laboratory
- Laboratory D**    UV-Visible Spectrometry Laboratory
- Laboratory E**    High performance Liquid Chromatography Laboratory
- Laboratory F**    Gas Chromatography/Mass Spectrometry Laboratory
- Laboratory G**    Fourier Transform Infrared and Raman Spectrometry Laboratory
- Laboratory H**    MALDI Time-of-flight Mass Spectrometry Laboratory (Note this is a one day lab that everyone will do the week of March 21, two groups on Tuesday (3/21) and two groups on Thursday (3/23))

**Note: The laboratory will be closed during the week of March 10 (Spring Break)**

Students will be divided up twice (January 17 and February 23) during the semester by random draw into four groups of 3 students. These will determine the order in which the various experiments are conducted (see attached schedule)

**Special Project Laboratory - Group Problem Solving Exercise:** Students will work on a special laboratory project during the last several weeks of the semester (2 weeks in lab, one week for writing the final project report, the off week is to be used for additional project planning). At the same time as the second group assignments are made (to be done on Feb. 23), you will also be assigned to one of three special project groups (composed of four students each). After the final project group assignments are made, your group should meet to discuss and plan your group experiment (see details below). The last three weeks of the semester is reserved for the final group project. **Lab closes for experimentation on Thursday April 27th.** Group reports on the special project are due by April 28 at 5 PM in the Instructor's office (no late special reports accepted).

As a group, your special project team will devise an appropriate set of experimental procedures to address a problem of your choice (see examples below) using any of the experimental analytical instrumental techniques from this semester's laboratory. Any additional methods will need approval from the instructor (see Pre-Project Proposal). Each student must contribute to the problem's solution and contribute to the writing of a detailed group laboratory report covering the basics of the problem, the methods employed, the experimental procedure

followed and the experimental results obtained (either positive or negative). The report format should be similar to the regular laboratory report (see below), but should provide expanded sections covering experimental methods and a more detailed discussion and reference section (i.e., it should be longer given 4 students are contributing to it). The cover page should also include the names and signatures of the student's contributing to the report to certify that all group members participated significantly to the project.

**Pre-Project Proposal:** Your group is required to submit a short (2 pages suggested), written proposal, outlining the specifics of your group's special project. This proposal should state the goals of the project, required chemicals and instrumentation, a concise justification of your analytical approach, and a brief outline of your experimental plan. It is *strongly* recommended that your group consult with your instructor well before it finalizes its proposal to ensure that the approach is viable. The instructor must approve all pre-project proposals. This will also allow time to order any special analytical reagents or standards needed. The proposal must be submitted, no later than the beginning of the first Tuesday lab period after Spring Break. Any reagents required must be readily available from the Chem Stores stockroom or easily ordered (for nominal prices) from a chemical supply house and must not be restricted substances or chemical or biological hazards.

### **3 Example Special Projects (Groups of 3 or 4 students each) - Groups May Use A Similar Project If They Modify Them In A Substantial Way**

- 1) Select several (4 or 5) inorganic species likely to be in various water samples for analysis. Choose several water sources (tap water, Logan river water, bottled water, great salt lake water, irrigation water, etc.) and determine the concentration of the species chosen. (Major instrumental methods to include: AA and ion selective electrodes and pH for  $\text{H}_3\text{O}^+$  and  $\text{OH}^-$  levels). You could alternately choose river sediment, sea shells or (harder) plant materials. While involving additional sample preparation, a more ambitious project will be judged favorably as to final grades.
- 2) Analyze (qualitatively and quantitatively) various components of volatile fuels (lighter fluid, gasoline, etc.) including possibly those from several brands or sources. (Major instrumental methods to include: GC-MS and perhaps IR and UV spectrometry). Alternately, fragrances might be analyzed (or even volatile flavor samples, if an appropriate source can be found like essence of spearmint).
- 3) Analyze the (active or inactive) ingredients from several over the counter medicines (liquids or solids) as to amounts and compare different brands. (Major instrumental methods to include: HPLC and perhaps IR, Raman and UV spectrometry). Examples cough syrup, pain medication, etc.
- 4) Be creative, but make sure the task is doable.

**Final Note:**

**In accordance with the Americans with Disabilities Act, reasonable accommodation will be provided for all persons with disabilities in order to ensure equal participation in Chemistry 5650. A student who requires an accommodation must contact the Instructor. The disability must be documented by the Disability Resource Center. In cooperation with the Disability Resource Center, reasonable accommodation will be provided for students with disabilities. Course material may be requested in alternate formats through the Disability Resource Center.**

**Please note that because of University holidays, lab will not meet the week of February 21, as per University policy, students will follow Monday's class schedule.**

<u>Date of Lab</u>	<u>Group 1</u>	<u>Group 2</u>	<u>Group 3</u>	<u>Group 4</u>
1/24 & 1/26	Experiment A	Experiment B	Experiment C	Experiment D
1/31 & 2/2	Experiment B	Experiment C	Experiment D	Experiment A
2/7 & 2/9	Experiment C	Experiment D	Experiment A	Experiment B
2/14 & 2/16	Experiment D	Experiment A	Experiment B	Experiment C
2/21 & 2/23	No Lab 2/21	Instrument Overview (2/23) for Experiment E-H		
2/28 & 3/2	Experiment E	Experiment F	Experiment G	Open (Special)
3/7 & 3/9	Experiment F	Experiment G	Open (Special)	Experiment E
3/14 & 3/16	Spring Break Week - No Laboratories			
3/21 & 3/23	Experiment H	Experiment H	Experiment H	Experiment H
3/28 & 3/30	Experiment G	Open (Special)	Experiment E	Experiment F
4/4 & 4/6	Open (Special)	Experiment E	Experiment F	Experiment G
4/11 & 4/13	Open laboratory periods for special project laboratory work			
4/18 & 4/20	Open laboratory periods for special project laboratory work			
4/25 & 4/27	Work on Special Project group reports due by 5 PM on 4/28 in W 026			