CHEM 5650 - INSTRUMENTAL ANALYSIS LABORATORY - Spring 2019

Instructor: Dr. Robert S. Brown **Office:** Widtsoe 026
Phone: 797-0545 **E-mail:** bob.brown@usu.edu

Office Hours: Tuesday and Thursday 2:30-3:20 PM and by individual appointments

Laboratory Meetings: Tuesday/Thursday 8:30 – 11:30; ML 362 and ML 364
Reference Text: Principles of Instrumental Analysis; Skoog, Holler & Crouch, 6th/7th Ed.
Laboratory Procedures: Individual handouts provided for each of the laboratories

T.A.: Jenna Bouvang **email**: jenna.hawley@aggiemail.usu.edu

Laboratory Grading:

Specific Labs and associated Reports (8 @ 100 pts/report)	800 pts
Special Project Design & Formal Report	200 pts
Total Points	**1000 pts**

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: 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 that you need to solve and identify those chemical and physical properties of a sample that can be exploited to provide 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. **Approved safety eyewear and laboratory coats are to be worn at all times during the laboratory.**

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 preferably should have duplicate (carbon) numbered pages that can be removed and turned in to your instructor after every laboratory period in which data is collected. Alternately students can make photocopies of notebook pages to turn in by the end of the lab day. 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.**

Include the following information prior to the start of the laboratory period (Instructor/TA may check during the lab and deduct points if not completed).

Title of the experiment
Date experiment is to be conducted

Objective: Include a brief statement of the experimental goals.

During the laboratory period, make sure to record:

Laboratory Coworkers names for that day's laboratory

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

Always include details on the specific sample being analyzed (source, manufacturer, unknown number, etc.). This is critical for proper grading. Each student should individually analyze duplicate samples for any quantitative experiment and students should each analyze a different set of samples. Group members, as needed, may share common reagents or calibration standards needed in a specific laboratory experiment. Each group member prior to use should confirm the proper preparation of such common reagents or calibration solutions (i.e., double check the preparation procedure).

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. These may be included in the data collected section (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) or include them as an appendix at the end of the lab information (i.e., after last recorded data). Original copies should also be included as an appendix with the submitted laboratory report.

Calculations: Include any pre-lab calculations and calculations performed during lab.

Note: Late laboratory reports may be accessed a penalty (up to 10 points/week late), at the discretion of the Instructor and TA.

Laboratory Fees: A laboratory fee ($75) is required for this course. The laboratory fee is used to pay for reagents, help maintain the instrumentation and glassware and covers a small portion of the lab Teaching Assistant's support.

List of Chem 5650 Instrumental Analysis Laboratories for Spring 2018

Typically 2 or 3 students per group (see the lab rotation schedule provided)

Laboratory A Electronics Laboratory

Laboratory B Ion Selective Electrode Fluoride in Water Laboratory

Laboratory C Atomic Absorption 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 Spectrometry Laboratory

Laboratory H MALDI-MS + NMR Laboratory

Note: The laboratory will be closed the week of March 11-15 for Spring Break

Students will be divided during the first laboratory meeting (January 10) into three groups of 2 students each. This will determine the order in which the various experiments are conducted (see attached schedule). After experiments (A-D) are completed, group composition may be shuffled, at the instructor's discretion.

Special Project Laboratory - Group Problem Solving Exercise: Students will work on a special laboratory project during the last several weeks of the semester. This will include 2 scheduled weeks working in the lab on the project and 1 week for writing the final project report and final lab cleanup. An open unscheduled lab week (see attached schedule and is subject to change if required) is provided for all groups to facilitate project planning. This year, there will be 2 or 3 special project groups composed of 2-3 students. You and your group should begin meeting to discuss and plan your group experiment (see details below) early in the semester. The last 3 (or 4 if needed) weeks of the semester are reserved for the final group project (setting up, performing experiments, cleaning up the lab and writing the group project report). **The Lab closes for experimentation on Thursday April 18th (if needed). Mandatory final lab clean up is to be completed no later than Tuesday April 23rd**. Group reports on the special project are due by April 26 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 project's goals 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 full 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 proportionally longer given that multiple 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. Early discussions will also allow time to order any special analytical reagents or standards that may be needed. The group pre-project proposal must be submitted no later than **Friday, February 29** (two weeks prior to spring break!). Any reagents that will be required must be readily available from the Chem Stores stockroom or easily ordered (maximum $100 budget for supplies) from a chemical supply house and must not include restricted substances or chemical or biological hazards.

Example Special Projects (Groups consist of 2 or 3 students, depending on class size).

Note: Groups may choose a similar project if the project goal and scope is different in a substantial way and the groups work independently. However, instrument time-sharing may be a problem and this must be worked out satisfactorily among the project groups prior to project approval.

1) Select several (4 or 5) inorganic/organic 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 H_3O^+ and OH^- levels, etc.). You could alternately choose river sediment, seashells or (harder) plant materials. While involving additional sample preparation, a more ambitious project will be judged favorably as to final grades. Project should include optimization and examination of interferences.

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, determine the minor volatile components of a flavor (such as peppermint oil, available from grocery store) or fragrance (such as a perfume). Also, could explore a synthesis product and identify major and minor components.

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. Any inorganic trace heavy metals present?

4) Determine the capsaicin content of various chili peppers using HPLC. Compare capsaicin content that you determine to the Scoville hot scale (for reference see: http://en.wikipedia.org/wiki/Scoville_scale), which is a subjective scale based upon the perceived hotness of human taste testers. The group could also subjectively rank the chilies as to hotness by taste testing them outside of the laboratory.

Note: Be creative, but make sure the project is achievable within the given time and budget (maximum $100 will be provided for the purchase of necessary chemicals or supplies).

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, there will be no lab scheduled for February 21. As per University policy, students will follow Monday's class schedule on Tuesday February 20. There will be a dry lab (MALDI-MS) on Thursday February 22. Note also, special project groups should utilize open lab periods to discuss and begin planning for the group's special project.