CHEMISTRY 311 "W"
ADVANCED ANALYTICAL CHEMISTRY
SPRING 2009

INSTRUCTOR

Duane Weisshaar Office: GSC 254 e-mail: duane.weisshaar@augie.edu
Phone 274-4812 Home: 339-7269 website: faculty.augie.edu/~chem
Office Hours: 9-10 MTWF or by appointment

CLASS SCHEDULE

Lecture - Discussion	11:00 - 11:50 MW	GSC 241
Lab	2:00-4:50 pm T & 8:00-10:50 am R	GSC 259

TEXTS and SUPPLIES

Skoog, D. A.; Holler, F. J.; Nieman, T. A. *Principles of Instrumental Analysis*, 6th ed., Saunders College Publishing: Fort Worth, 2007.
Dodd, Janet S. *The ACS Style Guide*, 2nd or 3nd ed., American Chemical Society: Washington, DC, 1997 or 2007.
Bound Lab Notebook with carbon or carbonless pages
Safety Goggles

Grading Criteria

POSSIBLE POINTS		**APPROXIMATE CUTOFFS**	
Exams (3)	300	A - A-	930 - 900 + A Candidacy Threshold
Candidacy Exam	20	B+ - B - B-	870 - 830 - 800 + B Candidacy Threshold
Lab Reports	400	C+ - C - C-	770 - 680 - 650
Instrument Proposal	100	D+ - D - D-	630 - 580 - 550
Review of Instrument Proposal	30	All +/- designations assigned on a case by case basis.	
Departmental Seminar	50		
Final Exam (ACS)	100	Late penalty assessments are listed with assignment descriptions and summarized on the last page of the syllabus.	
TOTAL	**1000**		

NATURE AND GOALS OF THE COURSE

This course deals with advanced topics in analytical chemistry with an emphasis on instrumental methods. The lecture portion of the course will address the underlying principles of a variety of instrumental techniques along with their strengths and limitations. You should be able to identify major components of instruments (block diagrams), discuss instrumental operation of each component, and work problems relating to the application of the instrument. In the laboratory you will explore topics of your choice in greater depth and gain practical experience utilizing the instruments available in the department to analyze particular chemical samples.

GOALS OF THE COURSE

- Survey instrumental methods of chemical analysis with an emphasis on the underlying principles as well as method strengths and weaknesses.
- Develop a scientific method of inquiry through problem-based lab exercises.
- Provide hands-on experience with chemical instrumentation for a quantitative determination and with the subsequent data analysis.
- Develop and refine oral presentation skills with an emphasis on the style expected for presentations at professional chemistry meetings.
- Develop and refine writing skills consistent with the goals set forth for a "W" component course (see below) with an emphasis on the style and format expected in the field of analytical chemistry.

This course is also a "W" component course - that means:

- The instructor has attended a "writing across the curriculum" workshop to assure consistency for "W" courses.
- The "W" course will provide **at least three assignments** for which instruction in the writing process will be given, and each course will require **at least fifteen pages of written work** from among the following sorts of activities:
  - brief essays or themes
  - an empirical or library research report
  - journaling
  - essay exam questions

- The writing assignments will help students think more clearly and become more effective learners and communicators in chemistry.
- Specific feedback to each student is provided during the writing process, including at least one draft-revision assignment.

In this course the writing component will be composed of four aspects:

- one major paper
- the lab notebook
- lab reports
- essay exams

On most assignments you may "win back" points missed by turning in a revision (more details given below). To pass the course you must make a reasonable writing effort in all these areas.

During the first lab period we will discuss the writing process, some of the specifics of writing in Chemistry, and the writing resources available in the Chemistry Department. The Writing Center, Humanities 220, will also provide assistance - contact them by email at writing@augie.edu or see more details about the Center (including hours) on line at http://www.augie.edu/dept/acad_services/writ_cntr/index.html.

HONOR CODE

As a community of scholars, the students and faculty at Augustana College commit to the highest standards of excellence by mutually embracing an Honor Code. As a College of the Evangelical Lutheran Church in America, we understand the individual and collective responsibility we have in fostering integrity. Ultimately, our purpose is to be an engaged body of academically excellent, highly articulate, and morally centered persons who learn about and examine the world together. We believe that only when we are honest with each other and ourselves can we begin to contribute to the world in a meaningful manner. To that end we pledge that we will abide by the highest standards of **academic integrity**, and that we will abide by the decisions of the joint student/faculty Honor Board. (The full description of the Honor Code is available at www.augie.edu/admin/acadaff/.)

What does **Academic Integrity** mean in this course?

- You do your own work on individual assignments (not copying others). On group assignments you contribute to the group effort and strive to understand all parts of the project, not just the part you do.
- In lab you are "true" to your data - your report reflects what *you* measured and observed; data is not changed or manufactured to fit expectations. If you missed collecting some data, see the instructor; don't copy someone else's data.
- Give credit where credit is due. When you gather data from the Web, books, magazines, etc. cite the reference (author, title, etc.).

I presume we are in this class to help each other learn some chemistry (yes, instructors learn in this class too), so I trust you to turn in work that reflects your efforts and also, that as individuals and in your small groups, to help each other adhere to the **Honor Code**. As a reminder of that commitment, when submitting a group assignment, each member is to **sign** it. In addition, the following statement will be appended to each exam:

> On my honor, I pledge that I have upheld the Honor Code, and that the work I have done on this assignment has been honest, and that the work of others in this class has, to the best of my knowledge, been honest as well.
>
> Signed _____

If you cannot, in good conscience, sign this pledge or an assignment or if you have other concerns about academic integrity in this course, please come visit with me (in confidence of course) or send me an e-mail note. At a minimum, students caught violating this code will receive a zero (0) on the assignment or exam and the incident will be reported to the Academic Dean in accordance with the Honor Code procedures.

ACCOMMODATING DISABILITIES AND SPECIAL SITUATIONS

Any students who feel that they may need to discuss reasonable accommodations for a disability in this course are encouraged to speak with the instructor as soon as possible. Students with questions regarding disability services including appropriate documentation and coordination of reasonable accommodations should contact Susan Bies at the Student Academic Support Service Office, Career Center Suite, Room 100, in the Edith Mortenson Center, 274-5503, susan.bies@augie.edu.

Chemistry, by its very nature, involves handling of potentially hazardous substances. The labs in this course will teach you how to properly handle these substances while minimizing the range of exposure. However, exposure effects vary from person to person. So, if you have asthma, allergies, are pregnant, or have other special circumstances, please inform the instructor so we can plan appropriate accommodations for your safety.

COMMUNICATIONS WITH THE CLASS

Please check your e-mail daily; many general announcements come via e-mail, and in this class it will be used for communication between student and instructor (hopefully between students too). The instructor will be using your @ole addresses compiled by the registrar. If you use another e-mail account, put a forwarder in your @ole account (contact the Help Desk for help in setting up a forwarder).

MOODLE

The instructor is experimenting with Moodle this semester and will attempt to have Powerpoint presentations used in class, answer keys to exams, and other information files for this class posted there. To make sure everything is accessible to students, that information will also be post on K:\chemistry\311\. It appears that Moodle's new gradebook will work for this class. If you encounter problems with Moodle, please let the instructor know.

Logging in:

Go to http://moodle.augie.edu (from the Augie homepage, click "Students" and then "Moodle")

- Log in using your Novell username (for example, mjsmith07). If this is your first time using Moodle, your Augie ID is your password. Please change your password - click on a your name in the upper right corner of the screen.
- If your instructor has already added you to the Moodle course, you will see your course listed on the left. Click the course name to enter the course. If you don't see this course, let the instructor know.

Troubleshooting:

Most browsers are set to block file downloads. If you find that when you click a link, nothing happens, chances are your browser is preventing file downloads (even viewing some files is seen by the browser as a file download.) To override this security feature, right-click on the pale yellow security warning that appears in the upper part of the screen and select "Download file". Then, go back and try the link again. This time it should give you the typical file download dialogue box (with choices of "Open", "Save", and "Cancel".) Clicking "Open" should let you see the file.

Many browsers are set to prevent pop-ups. But, Moodle uses pop-ups for discussion forum posts. If you find that when you click a discussion post, nothing happens, chances are your browser is preventing it from popping-up. To override this feature, hold the CTRL key down while you click the discussion posting.

If you have any technical problems with Moodle, contact **Sharon Gray**, Augie's Instructional Technologist. Her e-mail is gray@augie.edu. Her phone numbers are: 605-624-4907 (office), 605-624-8833 (home), and 605-670-0185 (cell).

ATTENDANCE

Attendance is required for examinations and laboratory work. Although attendance at lectures is expected, it is not required. However, you are responsible for all lecture material and announcements, whether present or not. If an absence is unavoidable for lab or an exam, notify the instructor as soon as possible.

BIBLIOGRAPHY OF RESOURCES FOR THIS CLASS

Several texts, lab manuals, and other resources will be available in the Seminar Room (GSC 262) on a set of shelves labeled Advanced Analysis between the blackboard and window. DO NOT REMOVE THESE REFERENCES FROM THE SEMINAR ROOM. Other resources available in the department are summarized below. These should be useful as supplements to your text, as resources for preparing reports, and as sources of experimental procedures.

References for Lab Experiments

- Several lab manuals on the shelves between the blackboard and windows
- Materials and directions from the instructor - includes procedures worked up in past offerings for this course and a variety of Applications Briefs from manufacturers (2 large binders on Adv Anal shelf).
- Articles in *Journal of Chemical Education*. A computer searchable index is available on line at

http://jchemed.chem.wisc.edu/journal/; select Search JCE Index in left panel.
- *Analytical Chemistry* - some of the studies presented there could be adapted for this lab (now in main library).
- *EPA's Sampling and Analysis Methods* - TD 423 .C65 1991 (Sem Room).
- *Official Methods of Analysis of the AOAC* - q QD79 .A73 1990 vol 1 & 2 (Sem Room).
- *Standard Methods for the Examination of Water and Wastewater* - QD142 .A5 1971 and 1985 (Sem Room).

Other General References on analytical chemistry in the Seminar Room or on-line.
- *Analytical Chemistry by Open Learning* series - Advanced Analysis shelves.
- Instrumental references compiled by the instructor (2 large binders on Adv Anal shelf). Table of contents for this is provided in a handout).
- *Journal of Chemical Education.*
- *Mikkelsen Library electronic resources* including all the ACS journals and a variety of others.
  - http://www.augie.edu/library/articles/index.html, explore other tabs in this menu as well.
  - Some may require a password - contact Lisa Brunick (lisa.brunick@augie.edu), Reference Librarian.
- The website for this course at http://faculty.augie.edu/~dew provides the syllabus, web versions of most of the handouts, links to some analytical chemistry sites, and old exams.
- Digital Library for the Analytical Sciences at http://www.asdlib.org is a rich source of material for all aspects of this course.
- SciFinder Scholar, a chemistry literature search engine, is accessible from most of the computers in GSC 240 and Mikkelsen Library. See the handout for details about using SciFinder.
- Scirus is another web-based chemistry search engine at http://www.scirus.com/.

References on Safety are located on the Safety Shelf in the Seminar Room. MSDS are also available via the World Wide Web; a selection of links to safety sites are available from the Augustana Chemistry Home Page at http://ed.augie.edu/~chem.

References on writing are listed in the Writing Process handout.

LECTURE & EXAMS

Lecture will rely primarily on the text book with some supplemental material supplied by the instructor. The lecture/exam schedule and outline of the text material to be covered are given at the end of this syllabus. **Exams** (3 at 100 points each) will cover the topics discussed in class up to that point. These exams will be open notes, open book exams with time limited to one class period. Exams typically include a "group question" where groups of 3-4 discuss the question for 10-15 minutes and then each person writes an answer. Since exams are part of the "W" component, students will have the opportunity to submit revisions on exams. Exam corrections are due one week after exams are returned to the class. The **final exam**, the ACS standardize Instrumental Methods exam, will be given at the scheduled time. The final is a "one-shot," multiple choice exam - there is no opportunity for revisions.

CANDIDACY

There are **TWO** criteria for attaining a grade in the A range or the B range in this course:

1. Attain the designated level of performance on the candidacy exam.
   AND
2. Achieve the corresponding cumulative percentage on exams and other assignments.

In other words, attaining candidacy (see criteria below) gives you a hunting license for (candidacy for) a grade in the A range or the B range, but does not guarantee that grade. **However, failure to attain candidacy at a particular level means that you cannot receive that grade no matter how well you do on exams and other assignments.**

The first opportunity for Candidacy is an ACS standardized exam in Quantitative Analysis (final for Chem 242) during the first month of the course (specific dates in the schedule on last page):
- This provides a review of concepts covered in Analysis, which will not be covered in class.
- The exam has 50 questions with a 110 min time limit.
- It may be taken at any time outside of lecture and lab.
- You may take this exam as many times as you want, but no more than once per day.
- The instructor is willing to help in your preparation for this exam by answering your questions about the concepts, but he will not discuss specific questions from the exam.
- To encourage you to take it early, the score required for candidacy at a particular level increases by week as

shown in Table 1.

Table 1. Designated Proficiency Levels for Candidacy				
Target Grade	1st Week	2nd Week	3rd Week	4th Week
	Pctile	Pctile	Pctile	Pctile
For B Candidacy	57	61	64	68
For A Candidacy	80	83	87	90
Percentile indicates the percentage of the pool that scored **lower**				

- As soon as you establish candidacy for the grade of your choice, you may stop taking the exam.
- Once you establish candidacy for an A, you must stop.
- Points for the Candidacy Exam are based on the highest percentile achieved and are determined by the following formulas where % = highest percentile scored and %min = minimum percentile for that attempt at the specified grade level:
  - If A candidate, Points = [90 + (% - %min)/2] * 20
  - If B candidate, Points = [80 + (% - %min)/2] * 20
- Points for scores less than B candidacy are set by the final exam scale used in Analysis (Final Exam Score (100) = 0.573 * percentile + 38.2).

The final exam may also be used to establish candidacy, but candidacy exam points are determined by your best performance on the Quantitative Analysis exam. Candidacy targets for the final exam are shown in the table below.

	Pctile
For B Candidacy	60
For A Candidacy	80

DEPARTMENTAL SEMINAR (50 pts)

Chemists depend on oral presentation skills as much as they do on writing skills. To give you an opportunity to develop those skills, the Department has introduced an oral presentation component into all advanced courses, which is coupled with the Departmental Seminar Program.

- Students in advanced courses and research are obligated to give **one** seminar that semester. If you are in more than one such "activity," get together with the faculty involved to decide how you can meet the seminar requirement in all the areas with your one seminar.
- Preparation
  - Individually or lab groups
  - 20 minute presentation (15 min talk, 5 min questions) with appropriate slides or overheads on an analytical topic of your choice.
  - At least one week before the seminar is to be presented, submit a draft of the slides you plan to use to the instructor (email in Presentations, Powerpoint, pdf, or html format) at least one week before the seminar is to be presented.
  - Follow the recommendations in the *Oral Report Guidelines*.
  - Preparation of presentations, like writing, requires several revisions and fine tuning, and benefits from others' input. Early submission to the instructor encourages this process.
  - Check out the technology ahead of time to make sure your presentation work and you know how to run everything - be prepared.
- Presentation
  - The instructor will coordinate scheduling of the Seminar. For efficiency two presentations may be scheduled for the same day.
- Learning by Observation - developing oral presentation skills includes learning from others
  - Attend colleagues seminars and provide feedback (instructor supplies a form).
- Final "report"
  - Submit an electronic copy of the final draft of the slides by the first class day after your seminar. Make sure the title includes "Sem Final" and your initials.
  - Submit evaluation forms of colleagues seminars to the instructor by the first class day after the seminar. Instructor will provide your comments anonymously to the presenter.
- The 50 points are distributed according to the *Criteria* handout. 5% per day late penalty for both deadlines.

GENERAL GUIDELINES FOR WRITTEN ASSIGNMENTS

Good writing takes time and several revision drafts so these reports should not be left to the last minute for preparation. Part of the revision process is built into the various Reports as described in the sections below. Further details on the writing process and specifics on the writing assignments are provided below or in separate handouts.

- Reports should conform to the accepted style for papers in chemistry (see the *ACS Style Guide* and the *Writing Process* handout). Use articles in *Analytical Chemistry* as examples of what good reports should be like.
- The reports are to be typed and double spaced with one inch margins on all sides. You may pencil in some last minute revisions, but major revisions should be typed.
- Submit electronic and hard copy of each report. To help the instructor sorting out email attachments, make sure the file name(s) include an assignment identifier, version number, and all partners initials (e.g. for exptl and safety on an HPLC lab - E&S LC2 DEW.wpd).
- Include hard copy of *all* previous drafts with instructor's comments with each submission, with the most recent on top.
- Revisions are due one week after the graded report or exam is returned to you; late penalty - 1st week 50% of "recoverable" points, revisions more than one week late will not be accepted.
- The number of points possible on a revision is half the points "uncollected" on the previous submission, excluding points lost for accuracy and precision.
- Use grammar/style and spell checkers on later drafts to help with construction and clarification. Remember you may have to teach your checkers some chemistry.
- When you revise a document, address more than the specific comments made by the instructor or reviewer; look at how the revisions fit into the context of the document. Revising one part of the document may prompt changes in other parts of the document to maintain coherence.
- The instructor is also available for consultation on preparing reports. "Free" revisions are available on any report if you turn in a draft early enough to give the instructor time to "grade" it and return it before the deadline. The last version turned in by the deadline will be the one that gets graded. "Free" revisions are limited only by the time necessary for "turnaround" which is affected by the other demands on the instructor's time.

INSTRUMENT PROPOSAL (100 pts for report and 30 pts for the Review)

In a career most chemists will face two major types of "writing assignments," the technical report and the proposal. All of you have had some experience writing technical reports in the form of progress reports for research or formal reports for other chemistry courses. In addition the reports you write for labs in this course will give you more practice in some of the aspects of writing a technical report (more on this later).

This assignment will give you experience in the other major type - writing a proposal. The process for generating this Instrument Proposal is designed to simulate the way proposals are usually generated, i.e. a mixture of individual and group effort that involves several rounds of revisions and at least one round of Peer Review.

- The stages for generating the proposal are outlined in the *Proposal Guide* handout.
- The due dates for each stage are given on the last page of the syllabus.
- **Turn in all hard copies with instructor's or reviewer's comments from previous stages along with the recent draft**, and an electronic copy of the recent version. Make sure the file name includes an assignment identifier, version number, and all partners initials (e.g. prop1 DEW.wpd)
- There are no points allotted for Steps 1-3 (generating the draft submitted for review). However, these are essential steps in the overall process, so a late penalty of 5% of project points per week will be assessed for Stage 1 and 2.
- *The deadlines for Stages 3 and 4 are absolute*; turn in what you have. Submission of the draft for review (Step 3) and the review itself (Step 4) affect other's progress, not just yours, so it is imperative that they be turned in on time.
- Step 5 - 100 points are allotted for the Final Draft, distributed according to the *Criteria* handout. 5% per week late penalty.

LAB

This course has a heavier emphasis on lab and a correspondingly lighter emphasis on lecture than most chemistry courses. The 400 points for lab are prorated to the lab projects according to the hours spent on each (details below).

Lab will focus on two major goals

- Provide experience on instruments, especially instruments you have not used before, with an emphasis on quantitative techniques.
- Provide experience in the scientific method of inquiry through problem-based lab exercises (research-like projects), including literature searching for background, choosing an appropriate method, experimental design including a safety plan, adapting published procedures to fit your situation, data analysis, reporting results in an appropriate format. Details on these aspects are presented below.
  - A handout highlights some projects that are of particular interest to the Chemistry Department.
  - Projects related to research interests are also encouraged.
  - A listing of procedure references in a variety of categories compiled by the instructor are available on the Advanced Analysis shelves in the Seminar Room. The instructor has copies of most of the papers referenced.
  - If successfully completed, consider presentation at a South Dakota Academy of Science meeting and/or the Augustana Symposium.

Scheduled lab time expectations

All experimental activity must be carried out during the scheduled lab time. You are expected to log a minimum of 70 hours in the lab. This semester there are 81 hours of scheduled lab time, so you have some flexibility in scheduling. In your lab notebook, keep a table of the total hours logged for each experiment and a running total of hours logged on all labs.

- *Logable activities (carried out during scheduled lab time):*
  - up to 3 hours of planning time per experiment (except electronics experiment). Logged planning time must be **time spent during the scheduled lab time**, planning time spent outside of scheduled lab time is **not** logable. Planning time during scheduled lab time beyond the 3 hours is lost lab time.
  - solution prep • instrument setup • instrument training with instructor
  - data collection • troubleshooting • cleanup
  - up to 2 hours data analysis time per experiment (except electronics experiment). Logged data analysis time must be **time spent during the scheduled lab time**, data analysis time spent outside of scheduled lab time is **not** logable. Data analysis time during scheduled lab time beyond the 2 hours is lost lab time.

General lab guidelines

Chemistry is a lab science, but it involves more than following a procedure and getting good results. The lab will give you experience in project design, attention to details, and 'covering all the bases" as well as execution. You will work in pairs on the experiments. Groups are allowed to select projects of personal interest within the following guidelines:

- All groups will do the electronics lab (follow the procedure provided by instructor) and at least two other projects.
- All other projects must have an analytical chemistry emphasis. Strive to include instrumentation you do not have experience with.
- One experiment must involve data acquisition and computer control with Labtech Notebook, Microlab, or other suitable interface package (not a canned package that comes with a computer-controlled instrument). The electronics lab should be completed before beginning this one.
- Notify the instructor of intended instrument use at least 1 *week ahead of time* to minimize conflicts with other groups and other classes. "First to sign up gets it" will be the general rule.
- Exceptions to these guidelines may be negotiated with the instructor.

Planning the experiment

- Not required for the electronics experiment
- Choose a problem and a method for solving - consult with the instructor to establish feasibility and refine your choice.
- Conduct a literature search to discover background material on your problem.
  - Include a SciFinder computer search. You may also include searches of other computer databases like: the WEB, *J. Chem. Ed.* index (on the WEB), Scirus, and the instructor's personal database.
  - Other sources that may be helpful include:
    - *Official Methods of Analysis of the AOAC, Standard Methods for the Examination of Water and*

Wastewater,
    - *Analytical Chemistry* especially the "Annual Reviews" issue (June 15 issue each year, alternates between applications and fundamentals).
  - Evaluate resources for credibility, especially Web resources. Remember, anyone can put anything they want on the Web; there are no controls to guarantee scientific accuracy or even a scientific basis.
- Write a draft of the Introduction/Background section of the final report. (paragraph form preferred, but detailed outline accepted) Include:
  - a clear statement of the problem you will investigate
  - background from work already done (from the literature search).
  - the rationale for the study and how past work relates to what you want to do
  - the basis of the method if it is not a standard method.
  - references used (ACS format, endnote or footnote).
- Write out draft of the Experimental section. (paragraph form preferred, but detailed outline accepted) Include:
  - a list of the equipment and reagents (purity and mfgr) you will need and an outline of the specific procedures you will use (plan of attack). This will help the instructor determine the feasibility of your method.
  - the method you will use
  - detailed procedures (to the extent possible), use of standard to verify things are working, initial instrument parameters, use of controls and unknowns, sample collection, etc.
- Write a Safety Report. For each reagent, identify:
  - the hazards to watch for as you use it.
  - how you will handle it.
  - how you will dispose of it.
- Submit the Planning Report (hard copy and email) with the Intro, Safety, and Experimental sections. Make sure the file name includes 311, proj id, draft #, and partners initials. Meet with the instructor for final approval before you begin work in the lab. Be prepared to explain what you plan to do and why.
- Once your plan is approved, schedule with the instructor to get checked out on any instruments, unless all group members are certified on that instrument.
  - Suggestion: During the first week select all the projects you plan to do and begin working on the Planning Report early. Writing assignment deadlines pile up during the second half of the semester.
- The Planning Report will be returned with instructor comments. You will revise this section and incorporate it into your final report.

Executing the experiment

- *WEAR SAFETY GOGGLES AT ALL TIMES.*
- **Each person** keeps an up to date notebook.
- Explicitly plan data collection to ensure you get everything needed to complete the Results report.
- Consider carefully the quantity of solutions needed. Adjust the "recipe" accordingly; make only what you need.
- Think carefully about the purity of reagents that you will need, particularly for standards.
- **Label all containers** with contents, concentration, initials, and date.
- **Do not store solutions in volumetric flasks.**
- Follow the handling and disposal precautions you documented in your Safety Report. If you have questions or need special equipment, see the instructor. Reduce, reuse, and recycle whenever possible, but dispose of solutions that do not have a specific use in other experiments or courses. **You take care of everything you produce.**
- When appropriate post "In Progress" signs on the experiment and on the front door to the lab.
- Clean up your work area at the end of each lab period. Storage in the hoods is **not** acceptable, other classes need to use that space too.
- **AFTER EACH EXPERIMENT**: properly dispose of all reagents used, clean glassware and return it to the stockroom, put all equipment away. You will be **penalized up to 50 points** for any leftovers discovered after the last lab period. Remember cleanup is a logable activity.

Lab notebook

The laboratory notebook serves as **permanent**, **chronological** record of what has been done in lab. In legal matters, such as the dispute of results reported in papers, patent applications or court cases, the lab notebook is considered the authority on the matter. It is imperative that a chemist maintains a detailed written account of lab work. **Anyone in**

the field should be able to pick up the notebook and understand what is written there well enough to repeat the experiment or calculate the results.

EACH STUDENT will

- Maintain a **complete** record of the progress for each experiment.
- Turn in the carbon pages recorded since the last lab period at the end of each lab period. The instructor should, at any time, be able to find in your notebook the information necessary to reconstruct the progress of an experiment.
- Before the first laboratory experiment, **number consecutively all the right hand pages of your lab notebook**.
- Submit notebook carbon pages at the end of **each lab period**. Late penalty - no charge for first offense, 5% of the Final Report for each instance thereafter.

The notebook must include:

- Page 1 **Title page.** Includes course name, semester and year, your name, campus box or address, email address, instructor's name.
- Pages 2-4 **Table of Contents.** Entries must be in chronological order and should provide page numbers for pertinent data and calculations. Keep this current as the semester progresses.
- **Log of Hours Spent in Lab.** Keep a table of the total hours logged for each experiment and a running total of hours logged on all labs. Logable activities were outlined above.
- Remaining Pages **Data Entry.**
    - Flexible format.
    - Document enough information to enable someone not in your group to complete the Notebook and Results Report.
    - **Record all raw data in ink directly in the notebook unless it is collected via a computer interface.**
    - On the first notebook page for each experiment record:
        - The date, your name and your partner's name in the upper right hand corner.
        - Experiment title on the first line.
        - Complete bibliography used to develop the project procedure and describe any modifications used.
        - Unknown identification numbers.
    - Date and initials at the top of all other pages.
    - Record all numerical data with correct number of significant figures and units.
    - Observations included as primary data - what did you do and what did you see?
    - All instrumental parameters.
    - Cross-reference to printouts generated by an instrument that are stored elsewhere. These printouts should be inscribed with name, date and pertinent instrument parameters to facilitate interpretation.
    - At the bottom of the last page for each lab period list:
        - time logged for that day.
        - running total of time logged on that experiment so far.
        - running total of time logged in lab so far.
        - sign and date the entry.
- Whenever practical, use spreadsheets and other computer programs to compile the Data Analysis and Statistical Report (details below). An electronic copy of the report form is available on the class web site.

Notebook and results reports

- Submit the report (hard copy and email) one week after completion of the lab. Make sure the file name includes N&R, proj, draft, and partners initials.
    - For revisions, include hard copy of all previous drafts that include the instructor’s comments.
    - The electronics lab report consists *only* of the carbon pages from the notebook and the group’s answers to the questions.
- Show sample calculations.
- Instructor will provide feedback on calculations, accuracy, and precision.
- These results will also be merged into the Final Report, but not necessarily in this form.

Final report

- Revise the Introduction/Background, Safety, and Experimental sections turned in during the planning process.
- Add a Results and Discussion section that is appropriate for the project.

- Consult with the instructor to decide how to format this section. For some projects it will be in a journal format, for others it might be an instruction manual or other more appropriate format.
- Use appropriate figures and tables to present the results.
- Discuss the relevance of the results and in general relate them to the Introduction.

- Include a conclusion.
  - What did you learn from the experiment?
  - Include a discussion of "what's next?" What kinds of things still need to be addressed (things for someone else to do).
- References section
- These reports are due one week after completion of the lab. Work on your reports as you work, don't leave all the writing until the end.

****

GENERAL COURSE OUTLINE

GENERAL APPROACH

The lecture will concentrate on instrumentation and the theory of operation of instruments and their component parts with a major emphasis on Instrumental Signal Processing and the techniques of Chromatography, Voltammetry, and Spectroscopy. More time will be spent on HPLC and Voltammetry which are not covered to any great extent in other courses. The text will be the primary source of material, with some additional information supplied by the instructor or gleaned from the other resources available.

Students should concentrate on learning block diagrams and the major options available for each block, theory of operation for the instrument and for the various options for each block, and the basic electronic circuits used in each option for each instrument or technique. In addition the student should have some feeling for the performance characteristics (figures of merit) for each technique including: precision, sensitivity, detection limit, dynamic range, selectivity, and bias (these are not normally collected together in tables, they must be ferreted out of related discussions, several sources may be required to get a complete picture).

It is assumed that students have retained knowledge of the following concepts from past courses:

From Analysis:
Use of statistics: average, standard deviation, confidence interval (assume 95%), and linear least squares regression and associated statistics. Review - Appendix 1 this text, Analysis texts, Excel (Analysis lab manual or from the class website).

Redox: balancing reactions, Nernst equation and associated calculations. Review - Chap 22 and 23 this text, Analysis texts.

Acid-Base: pH, equilibrium calculations. Review - Analysis texts.

From Organic and Physical Chemistry:
Interpretation of NMR, IR, MS, UV/VIS of organics, x-ray diffraction spectra. Theory of absorption, emission, fluorescence, phosphorescence, x-ray diffraction, Raman scattering, and Beer's Law. Review - Chaps 6-19 this text, Physical Chemistry and Organic texts.

From Physics:
Basic electronics: Ohm's law, resistor networks, phase relationships in ac applications, simple electronic components and their schematic symbols. Review - Chap 2 this text. The electronics lab will also help you review.

CHAPTER OUTLINES

Chapter 1 - Introduction (pp 1-24)
Data Domains
Detectors, Transducers, Sensors
Selecting a Method (this section may be helpful for the Literature Assignment)
Calibration Methods (calibration curve, standard

addition)
Performance Characteristics: precision, bias, sensitivity, detection limit, concentration (dynamic) range, selectivity.

Chapter 2 - Electrical Components and Circuits (pp 26-58)
Reference and review material
Some key sections: voltage dividers, loading error, capacitors and capacitance, filters based on RC circuits, response of RC circuits to pulsed inputs

Chap 3 - Operational Amplifiers in Chemical Instrumentation (pp 59-79, handout)
Design circuits (draw schematics) to do specific tasks
Calculate the output for a given circuit.
Primary emphasis - follower, follower with gain, inverter/summer, difference/instrumentation amplifier, current follower, integrator.

Chapter 4 - Digital Electronics and Microcomputers (pp 80-109)
Analog and Digital Signals
Digital-to-Analog (DAC)
Analog-to-Digital (ADC) Converters
Analog to digital conversions: Nyquist sampling theorem - the sampling rate must be at least 2 times the signal frequency to prevent information loss. Sampling at a lower frequency can result in aliasing - recording of what appears to be a good signal, but at a lower frequency than the original. The sampling rate can also be used as a filter: sampling at 60 Hz will eliminate 60 Hz noise induced from power lines.

Chapter 5 - Signals and Noise (pp 110-125)
Signal/Noise ratio
Types of noise - sources and strategies to minimize
Noise reduction methods, their limitations and advantages: analog filters (RC), modulation (choppers), computer averaging methods (boxcar, Savistky-Golay, least squares, correlation, Fourier transform
Digital advantage: Noise increases a function of the distance the signal is transmitted. For analog signals, any noise is a direct degradation of the signal, and the signal is difficult if not impossible to recover. Noisy digital signals are relatively easy to recover as long as the noise does not cross the on/off threshold of the digital encoding. Hence, digital signals can be transmitted over much longer distances.

Chapter 6 - An Introduction to Spectrometric Methods (pp 132-163)
Reverence and review material

Chapter 7 - Components of Optical Instruments (pp 164-214)
Monochromators
Advantages of gratings over prisms
Radiation transducers (PMT, diode array, CCD, thermal)
Advantages of mirrors over lenses
Fourier Transform advantage

Chapter 8 - An Introduction to Optical Atomic Spectrometry (pp 215-229)
Line broadening mechanisms
Sample introduction methods

Chapter 9 - Atomic Absorption and Atomic Fluorescence Spectrometry (pp 230-253)
AA block diagrams and theory of operation
Background correction methods and theory
Advantages and limitations of flame vs furnace atomization/excitation background correction methods and theory
Advantages and limitations of Absorbance vs. Emission methods

Chapter 10 - Atomic Emission Spectrometry (pp 254-280)
ICP block diagrams and theory of operation

Chapter 11 - Atomic Mass Spectrometry (pp 281-302)
Block diagrams and theory of operation
(more on Mass Spec in Chapter 20)

Chapter 12 - Atomic X-ray Spectrometry (pp 303-331)
Block diagrams and theory of operation
Difference between x-ray fluorescence and x-ray diffraction

Chapter 13 - An Introduction to Ultraviolet/Visible Molecular Absorption Spectrometry (pp 336-366)
Block diagrams and theory of operation
Advantages and Disadvantages of single and double beam instruments

Chapter 14 - Applications of Ultraviolet/Visible Molecular Absorption Spectrometry (pp 367-398)
Photometric titrations

Chapter 15 - Molecular Luminescence Spectrometry (pp 399-429)
We will cover this chapter only if time permits.

Chapter 16 - An Introduction to Infrared Spectrometry (pp 430-434) and
Block diagrams and theory of operation

Chapter 17 - Applications of Infrared Spectrometry (pp

455-480)
Block diagrams and theory of operation

Chapter 18 - Raman Spectroscopy (pp 481-497)
Block diagrams and theory of operation

Chapter 19 - Nuclear Magnetic Resonance Spectroscopy (pp 498-549)
Block diagrams and theory of operation

Chapter 20 - Molecular Mass Spectrometry (pp 550-588)
Ionization sources theory and operation
Quadrupole
E & M sector
Ion trap
Tandem MS
Electrospray

Chapter 21 - Surface Characterization by Spectroscopy and Microscopy (pp 589-623)
Origin of ESCA and Auger (o-jay) signals
SEM and microprobe analysis
Block diagrams and theory of operation
STM and AFM

Chapter 22 - Introduction to Electroanalytical Chemistry (pp 628-658)
Double layer theory
Modes of mass transport
iR drop
Overvoltage

Chapter 23 - Potentiometry (pp 659-696)
Reference and review material

Chapter 24 - Coulometry (pp 697-715)
Reference and review material

Chapter 25 - Voltammetry (pp 716-756)
Compare and contrast the input (voltage) and output (current) signal for methods
Differential pulse polarography (DPP)
Square wave voltammetry
Linear sweep voltammetry and cyclic voltammetry
Potentiostat
Charging and Faradaic current
i - E and i - C profiles - macro and micro electrodes
Reversible and irreversible systems (text does not clearly distinguish between chemical and electrochemical definitions)
Block diagram and theory of operation

Chapter 26 - An Introduction to Chromatographic Separations (pp 762-787)
Separation modes
Definitions and formulas for calculation: retention time, capacity factor, selectivity, plate height, theoretical plates, resolution
van Deempter equation: terms and conditions to optimize each comparison of optimum conditions for lc vs gc

Chapter 27 - Gas Chromatography (pp 788-815)
Block diagram and theory of operation
TCD
FID
MS detector

Chapter 28 - High-Performance Liquid Chromatography (pp 816-855)
Block diagram and theory of operation
Types: normal phase, reverse phase, ion, size exclusion
Detectors - RI, UV, fluorescence, electrochemical, ELS

Chapter 30 - Capillary Electrophoresis, Capillary Electrochromatography, and Field Flow Fractionation
Electrophoresis and Capillary Electrophoresis

Chapter 29, 31-34 -
Reference material, we will cover these if we have time.

CHEM 311 SPRING 2009 TENTATIVE LECTURE SCHEDULE			
Mo	Days	Topic	Chapters
Feb	9	Introduction	1
	11, 16	Operational Amplifiers	2, 3
	18	Digital Electronics	4
	23, 25	Signals and Noise	5
Mar	2	Intro to Electroanalytical Chemistry	22
	4, 9	Voltammetry	25
Exam 1 Mar 11 (W)			
	16	Introduction to Chromatography	26
	18	Gas Chromatography	27
	23-27	**Spring Break**	
	30	High Performance Liquid Chromatography	28
Apr	1	Capillary Electrophoresis	30
	6	Components of Optical Instruments	7
Exam 2 Apr 8 (W)			
	10-13	**Easter Break**	
	15, 20	Atomic Absorbance and Emission Spectrometry	8, 9, 10, 11
	22	Introduction to Molecular Absorption Spectrometry	13, 14
	27	IR Absorption and Raman Spectrometry	16, 17
	29	NMR Spectrometry	18
May	4	Mass Spectrometry	20
Exam 3 May 6 (T)			
	11	X-Ray Spectrometry	12
	13	Surface Characterization	21
Final Exam May 18 (M) 8:00 am			

ASSIGNMENT DUE DATES		
DUE	ASSIGNMENT	LATE PENALTY
Before Starting Each Lab	Draft Planning Report	5%
After Each Lab Period	Notebook Carbons	1st Free, thereafter 5% each
1 Week Before Presentation	Draft of Presentation Slides	5%/day
1st Class Day After Seminar	Final Draft of Slides	5%/day
1 Week after Lab Completed	Revised Final Report and Notebook & Results	5% per week
1 Week after Returned	Revisions	1 week - 50% possible pts, >1 week - not accepted
Feb 5 - Mar 5	Candidacy Exam	no more chances until final
Thurs, Feb 12, 5 pm	Instrument Pre-Proposal	5% of formal
Wed, Mar 4, 5 pm	Proposal - 1st Draft	5% of formal
Mon, Apr 6, 5 pm	Proposal - Combined Draft	turn in what you have!
Tues, Apr 14, 5 pm	Proposal - Review	turn in what you have!
Mon, May 4, 5 pm	Proposal - Final Draft	5% of formal/week
Thurs, May 14, 5 pm	all revisions	not accepted
Thurs, May 14, 11:50 am	Completed Lab Clean Up	up to 50 results points
NO ASSIGNMENTS OR REVISIONS ACCEPTED AFTER 5 PM THURSDAY May 14 (LAST CLASS)		
ALL PREVIOUS DRAFTS MUST ACCOMPANY REVISIONS		

GRADING SUMMARY		
Hour Exams	100 pts each	as scheduled
Candidacy Exam	20 pts + establish candidacy	
Final Reports	percentage * (hrs/expt) * (400/total hrs)	
Departmental Seminar	50 pts	
Instrument Proposal	100 pts, score based on revision after review	up to half of pts back on final revision
Peer Review	30 pts	
Final Exam	100 pts	