Chemistry 333: Advanced Analytical Chemistry
Block 6, 2018-19

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Office hours: I'll be in the lab/classroom or my office most of the time between 8:15 and 5:00 each day. Please let me know if you would like to meet individually and we'll set a time.

Course Objectives and Meeting Times

Learning Objectives (Educational Priorities and Outcomes):

Students will:

1. Develop their understanding of modern methods of instrumental analysis, including spectroscopy, chromatography, mass spectrometry, and voltammetry (Knowledge, Inquiry)
2. Be able to describe the underlying principles, instrument hardware, and possible applications and limitations of each group of instrumental methods (Knowledge, Reasoning)
3. Apply new knowledge and careful reasoning to solve problems in the lab; present lab results with appropriate estimates of uncertainty (Inquiry, Reasoning, Ethical Behavior)
4. Search for relevant articles in the current literature and work as a group to understand them (Inquiry, Reasoning, Communication, Knowledge)
5. Practice communication skills through group work in the lab, writing clear and concise lab reports, and presenting the results of a lab project (Communication)

This course supports the Educational Priorities and Outcomes of Cornell College with emphases on knowledge, inquiry, reasoning, and communication.

Meeting times: Class will meet from 9:00 to 11:00 am and from 12:30 or 1:00 to 3:00 pm every day; see the schedule below. In general, mornings will be devoted to discussion and lecture, and afternoons will be spent in the lab. Friday afternoons will be reserved for catching up in the classroom and/or lab.

Course Materials, Expectations, Assignments, and Grading

T**ext:** ***Quantitative Chemical Analysis***, 9th edition, by Daniel C. Harris. You will also need a lab notebook with carbonless copies (a partial one is fine) and safety goggles.

Expectations for class participation: Arrive on time for class and lab. Read the text before class. Be prepared to answer questions about the readings in class. Contribute to the discussions; remember that questions are valuable contributions. On some days, individual students will take responsibility for leading the discussion of certain sections.

If you find it difficult to contribute to class discussions on the spot, please write out questions about the reading as you read, and bring your questions to class. I will try to incorporate the questions into class, and they will count as a contribution to the discussion.

Assignments:

1. Problem sets (Sapling) will be assigned on most class days. Some problems will be assigned before we discuss the material and can be completed as you read the text; others will be assigned after we discuss the topic.

2. Learning journal: Study each chapter using a variety of methods, and compile your work in a 3-ring binder. Here are some suggestions:

- Draw a diagram or flow chart about an instrument or concept. You can start from scratch or re-draw a diagram from the text or elsewhere to understand it better.
- Write summaries of sections of the chapter.
- Write your own description of how the instrument or concept works.
- Give a link to a website that you found helpful in understanding the topic, and describe what you learned. Manufacturers' websites often have excellent information, diagrams, videos, and applications of their instruments.
- Cite a journal article that makes significant use of the instrument or concepts, and write a paragraph about the research described.

Bring your learning journal to class each day; turn it in for grading at the midterm and on the last day of the block.

3. Research article summary and discussion: Choose a recent (within the last two years) research article on an application or development of one of the instrumental methods we are discussing; check with me to be sure it is appropriate. The journal Analytical Chemistry is a good starting point, but you are welcome to select an article from a different journal. If your article is longer than 6 pages, select approximately 6 pages of the article for the class to discuss. Write a ~3 page **summary** of the article and a list of **questions** (5-10) for discussion. Your summary should include the specific purpose of the research (why is it important?); the experimental approach; descriptions of sample preparation, data analysis, and/or instrumental details as appropriate; and the conclusions reached. Question topics might include experimental details (what kind of detector did the authors use, and why did they choose that detector?), data analysis (did the authors use a calibration curve, internal standard, standard addition . . .), interpretation of results (how do the authors justify this particular conclusion?), or just about anything else. Pay particular attention to the figures. Try to devise questions that require us to apply material we've discussed in class. All students will be responsible for reading all articles and participating in the discussions. After our discussion of the articles, turn in your marked-up copies.

4. Lab notebooks, reports, project plans, and project presentation: See below for more information.

Grading: Due to the descriptive nature of much of the material and the emphasis on lab, exams will carry less weight than in most chemistry courses, and more weight will be given to out-of-class assignments and class participation.

Problem sets and learning journal	16%
Midterm exam	20
Final exam	20
Lab reports, notebook, project plan, and presentation	28
Research article summary and discussion	8
Class participation	8

Grading scale: 93-100%=A, 90-92%=A-, 87-89%=B+, 83-86%=B, 80-82%=B-, 77-79%=C+, 73-76%=C, 70-72%=C-, 67-69%=D+, 63-66%=D, 60-62%=D-, <60%=F

The points possible for an assignment will be reduced by 10% for each day that the assignment is late, including weekend days.

Class Policies

Health issues: For your safety and the safety of those who will be working with you in the lab, please inform me and the lab instructor if you have a health issue that may be exacerbated by exposure to chemicals. Examples would be severe asthma, severe allergies, seizure disorder, or pregnancy. We will keep this information confidential and work with you to minimize your risk.

Academic Honesty expectations: Cornell College expects all members of the Cornell community to act with academic integrity. An important aspect of academic integrity is respecting the work of others. A student is expected to explicitly acknowledge ideas, claims, observations, or data of others, unless generally known. When a piece of work is submitted for credit, a student is asserting that the submission is her or his work unless there is a citation of a specific source. If there is no appropriate acknowledgement of sources, whether intended or not, this may constitute a violation of the College's requirement for honesty in academic work and may be treated as a case of academic dishonesty. The procedures regarding how the College deals with cases of academic dishonesty appear in The Catalogue, under the heading "Academic Honesty."

I encourage you to work together as you study the material and work the problems. Examples of inappropriate cooperation would be copying from another student's problem set or lab report, or sharing information during a test. Please be aware that these actions constitute academic dishonesty and will be handled in accordance with the policies in the student handbook.

Accommodations for learning disabilities: Cornell College makes reasonable accommodations for persons with disabilities. Students should notify the Coordinator of Academic Support and Advising and their course instructor of any disability related accommodations within the first three days of the term for which the accommodations are required, due to the fast pace of the block format. For more information on the documentation required to establish the need for accommodations and the process of requesting the accommodations, see http://www.cornellcollege.edu/academic-support-and-advising/disabilities/index.shtml.

Brooke Paulsen, the Coordinator of Academic Support and Advising, can be reached at bpaulsen@cornellcollege, 319-895-4382, or in room 309 of Cole Library.

Other policies: I turn off my cell phone when I come to class, and I expect you to do the same. It is not appropriate to text, go online, etc. during class or lab.

A student who wishes to drop the course on the 15th day must have completed all the work for the course and must have attended class faithfully.

Lab

Lab notebook: Since this is an advanced class, you will want to practice keeping an exemplary lab notebook. Create a Table of Contents on the first page. Turn in notebook page copies as you complete each experiment. For each experiment, your notebook should include:

- Title, date, and your name on each page
- Procedure reference, in American Chemical Society format (see below)
- Notes on deviations from the printed procedure. If you are developing a procedure, be sure it is written clearly in your notebook before you begin experimental work. Leave space below the procedure reference to modify the procedure later as you work.
- Data, in tabular form when possible. This is the most important part of your lab notebook. Be sure that the nature of the data (mass of what? volume of what?) and the units are indicated clearly.
- Observations; you do not need to narrate the lab, but do include any observations that will help you interpret data later on.

Lab reports: Submit a lab report for each experiment you perform. If you work as a member of a group, each member of the group will submit his/her own report. The audience for your lab report is a person who has taken a course in analytical chemistry and knows basic principles, concepts, and laboratory techniques, but is not familiar with the particular experiment. Lab reports must be word processed and must follow the format below.

> **Heading: Experiment Title, Student Name, and Date**
> **Method:** Write a brief paragraph describing the method used. Give a reference (in ACS format) to the printed procedure, and describe any deviations from the procedure. Your method need not include details such as volumes or concentrations, if they are provided

in the printed procedure. A competent chemist should be able to take your method section (including the procedure to which you refer) and reproduce what you did in the lab.

Reactions, if applicable: Include a balanced equation for each reaction studied. The reactions may be written by hand. (Not all of the experiments involve reactions.)

Data: Present the facts: the data and observations that you collected during lab. Arrange your data in tabular form, taking the essential data from your lab notebook. You may require more than one table; if so, clearly label each table with Table 1, Table 2, etc., and the nature of the table contents. You may include results in a table with your data if you wish.

Sample calculations: Show an example of each type of calculation performed. (Note: Carry an extra significant figure throughout your calculations to avoid rounding errors. Round to the appropriate number of sig figs when you reach the final result.) You do not need to show how to calculate a standard deviation. This section may be written in by hand.

Results and Discussion: In this section, you will use the facts presented in the data section to reach a conclusion (your results), and you will interpret your results for the reader. Clearly highlight your results; a person reading your report should be able to find your results quickly. It is acceptable to present the results in one table with the data, rather than in the Results and Discussion section. Be sure to include the following when applicable:

- The result of each trial (when multiple trials are involved) and a mean value with its uncertainty. The uncertainty will usually be the standard deviation or range. As in the data section, tables are often helpful for presenting quantities clearly. Don't forget units!
- Spectra and/or graphs with proper meaningful captions.
- If your result can be compared to a published value, then be sure to provide the published value and compare it to your value. Indicate where you obtained the published value by giving a reference in ACS format.
- Give your interpretation of your results. If your results did not agree well with the published value (where appropriate), what are the possible sources of error? As you write this section, ask yourself how confident you are in your results.

Take care to ensure that your report is written clearly and does not contain grammatical or spelling errors. Use American Chemical Society format for citing references, as outlined in *The ACS Style Guide: Effective Communication of Scientific Information* (3rd edition; Coghill, A.M.; Garson, L.R., Eds.; American Chemical Society: Washington, DC; Oxford University Press: Oxford, UK, New York, 2006). Chapter 14 of the style guide is available online at http://pubs.acs.org/userimages/ContentEditor/1246030496632/chapter14.pdf. The following website summarizes ACS style for a variety of sources: https://www.library.wisc.edu/chemistry/research-help/write-and-cite/acs-style-guide/ .

Lab experiments:

1. HPLC analysis of caffeine in beverages

2. Optimization of a separation by gas chromatography

3. Extraction, purification, and characterization of C_{60} from fullerene soot (UV-Vis, IR, and ^{13}C NMR) (two days)

4. Analysis of gourmet salts by ICP and AA

5. Electrochemistry: (1) Cyclic voltammetry
   (2) One of the following options:
   - Redox-linked reactions of acetaminophen
   - Detection of heavy metals by stripping voltammetry
   - Glucose biosensor
   - Prussian blue hydrogen peroxide sensor

6. X-ray fluorescence analysis of calcium in powdered milk. Prepare external standards or use standard addition method

Lab schedule:

Experiment	**M**	**T**	**W**	**Th**	**M**	**T**	**W**
HPLC	All						
GC/MS		CD		B			AE
C_{60} extraction		AB, E	CD				
C_{60} purification; IR and NMR			AB, E	CD			
ICP and AA				AE	BC, D		
Electrochemistry					AE	BD	C
XRF						A, CE	BD

Each letter corresponds to a person.

Class schedule (subject to change)

Day	Topic	Chapter	Assignment due
Week 1			
M	Analytical separations	23	
	Brainstorm lab projects		
T	Gas chromatography	24	
W	HPLC	25	
Th	Chromatographic methods and capillary electrophoresis	26	
F	Capillary electrophoresis	26	Lab report
	Literature searching for lab projects		
Week 2			
M	Mass spectrometry	22	Project plan -- brief
T	Quality assurance and cal. methods	5	Lab report
	Sample preparation	28	
W	Midterm		Learning journal
Th	Electrochemistry (review)	14	Lab project plan -- detailed
	Electrodes and sensors	15	
F	Electroanalytical techniques	17	Lab report
	Visiting speaker – Dr. Katie Brown '06		
Week 3			
M	Argonne		Lab report; submit journal article
T	PerkinElmer		
W	Spectrophotometry (review)	18	Lab report
	Applications of spectrophotometry	19	
Th	Spectrophotometers	20	Article summary and questions
F	Atomic spectroscopy	21	
Week 4			
M	AM and PM: Discussion of articles		Lab report; marked-up articles
T	Lab presentations		
W	Final exam		Learning journal; lab report