

Advanced Analytical Chemistry

Chemistry 440

Spring 2020

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Lecture:	TF 12:00 – 2 pm
Lab:	During class time and independently

Text: Skoog, Holler, and Nieman, *Principles of Instrumental Analysis*

Lab: Green, *Laboratory Manual to Accompany Quantitative Chemistry and Instrumental Analysis* and individual monographs

Curiosity has its own reason for existing. One cannot help but be in awe when he contemplates the mysteries of eternity, of life, of the marvelous structure of reality. It is enough if one tries merely to comprehend a little of this mystery every day. Never lose a holy curiosity.

- Albert Einstein

STUDENT LEARNING OUTCOMES The specific *Student Learning Outcomes* aligned to the *Chemistry Program Learning Outcomes* is that at the successful completion of this course participants should be able to successfully solve a variety of multi-step problems using mathematical and descriptive algorithms and, specifically in the laboratory portion of the course, participants will be able to utilize analytical chemical instrumentation properly including: preparation of high accuracy standards, set the operating parameters of different instruments, and perform calibration and analysis.

COURSE DESIGN AND CONTENT

To participate in this course you must have scored at least a C- grade in Instrumental Analysis (or the course equivalent at another university). Close familiarity with the following topics is necessary to excel in this course:

- ◆ Chemical equilibrium
- ◆ Separation science (e.g., HPLC, GC)
- ◆ Spectroscopy (UV-visible absorption and emission, FTIR, NMR)
- ◆ Mass spectrometry
- ◆ Electricity and magnetism
- ◆ Optics

The goal of this course is to introduce the participant to new techniques of analysis and to delve deeper into topics covered in earlier courses. The course is divided into 3 modules:

- ◆ Electrochemistry (potentiometry, voltammetry)
- ◆ Structural techniques (MS, NMR, FTIR)
- ◆ Other techniques not covered in earlier classes

In addition to lecture, readings, and laboratory work, a laboratory project is required as is a research paper. The project and research paper may not be on the same analytical technique or method. Specifics of these will be outlined later. The due date for the project and paper will be announced.

The overall goal of this course is to provide students with a sufficient understanding of the principles, laws, and theories of analytical chemistry to enable them to successfully analyze samples provided using selected instrumental methods. The student should gain the competence to follow a standard procedure, develop new procedures, operate chemical instrumentation in a safe manner, collect suitable data, evaluate the reliability of the data collected, and report the results in an appropriate form as would be required of any competent laboratory technician.

While the major foci are on the principles and capabilities of selected analytical instruments in addition to their design and construction, the specific goals are that every participant will...

- ✓ recognize and appreciate the value of analytical instruments used, as well as their limitations, in the solution of selected problems faced by the technician.
- ✓ realize the existence of a variety of instrumental methods, each with its own particular use based on its capabilities and limitations.
- ✓ understand that the selection of one instrumental method, as being superior to another in the solution of a particular analytical problem, is based on such factors as sensitivity, time required, selectivity, purchase cost of instruments involved, etc.
- ✓ understand that the success of any of the instrumental methods used depends upon a working knowledge and control of the operating parameters associated with each instrument studied.
- ✓ have a basic operational knowledge of the internal design of the instruments studied.

The theoretical background of many instruments will be given in lecture. Emphasis on the operating parameters associated with each of the instruments studied will be provided on a "need to know" basis. Operational details are covered sufficiently to provide an adequate understanding of the techniques used without being overwhelmed by minutia.

Upon successful completion of this course every course participant should be able to:

- ✓ understand the principles underlying the operation of each of the studied instruments and extrapolate that knowledge to other instrumental methods.
- ✓ describe the operational parameters for each of the analytical instruments studied.

- ✓ carry out calibration of instruments, preparation of a sample for analysis, safe handling of the sample during the analysis, and proper disposal of the sample after completion of the analysis.
- ✓ use techniques for recording and evaluating analytical data derived from instruments.
- ✓ solve a variety of numerical problems dealing with the analysis of samples using the various instrumental methods studied.
- ✓ appropriately report the results of a given analysis.
- ✓ write in scientific format reports of the theory, experimental method, and results of an analysis.

*We are what we consistently do; excellence...
therefore, it is not an act but a habit.*
- Aristotle

RELATIONSHIP TO THE SEAVER COLLEGE MISSION

From The Mission of Seaver College of Peppercorn University: *“Seaver College exists to provide a link between the knowledge and wisdom of the past and present with the challenges of the future. The college is essentially a community...[of] teachers committed to a life of instruction and scholarship [and] students preparing to assume responsible roles in contemporary society....”*

This course is designed to provide the framework on which hangs a significant portion of the body of basic chemistry knowledge, allowing the perceptive participant to glance into the richness of the microscopic world from a macroscopic point of view, and to provide the foundation for further studies in the sciences. Over the course of the semester, the successful participant will develop new and expand upon existing skills in critical thinking, mathematics, and the scientific method. Since chemistry is by its very nature an experimental science, honesty and integrity in the acquisition and analysis of data is at the very core of the scientific process. It is part of our role as practicing scientists to defend the nature of scientific discourse and to expose pseudoscience and scientific dishonesty.

ATTENDANCE Generally, roll will not be taken. However, since the content of exams and homework is often covered in lecture, missing class is not advised. Tardiness is disruptive – please try to be to class on time. If you *must* leave early, advise Dr. Green before class starts.

HOMEWORK Homework will be assigned as we go. Homework will be due on the assigned date. Late homework will not be accepted – please don’t ask. Much of the homework will not be graded. *You should do more than the assigned problems to master the material of this course.* You must keep up with the reading assignments. Do not put off reading the assigned material. Readings and lecture complement each other, they do not necessarily parallel each other.

QUIZZES Occasional unannounced short quizzes may be given during the semester so that you can track your progress and improve on deficiencies if necessary. If you miss a quiz for any reason, it cannot be made up.

EXAMINATIONS Two exams and a final examination will be given. A tentative exam schedule is given below. A test will be given approximately at the completion of each module. The final is cumulative. Unless there are extreme circumstances, no test may be

taken early or late. There is no mechanism built into this course to make up a missed exam. Make arrangements early if you will be missing an exam for participating in a university sponsored event. These events include research conferences, course fieldtrips, theater, athletics, etc. but not sorority or fraternity or nonsponsored events. In the event of an illness – with documentation from the University Health Center or other physician – contact the course instructor as is practical to make appropriate arrangements.

LABORATORY This course has no formal laboratory schedule. However, several laboratories are scheduled to illustrate concepts discussed. Some laboratory activities will be performed in lieu of lecture and *vice versa*. A laboratory project is required which will be discussed on an additional sheet.

GRADING **It is important to remember that grades are not wages.** You will not be graded on how hard you work – you will be graded on mastery of the assigned material. Your course grade is broken down as follows:

3 exams @ 100 pts.=		300 pts.
Homework	=	50
Labwork	=	100

		450

OFFICE HOURS Office hours are posted. Attending posted office hours are preferred but if you can't make a posted hour you can make an appointment or even just try dropping by. **Don't hesitate to get help if there's a problem.** If you are doing unassigned problems to gain proficiency, I will go over the problem with you if you wish. You may use e-mail to get non-immediate help as well. The professor's e-mail address is given above.

A Word On DEAD WEEK Contrary to common belief, the last week of classes is not reserved for review, partying, etc. There will be lecture on new material and homework will be due. There might be some final exam review during that week also. An exam is scheduled for the final week of class, as well.

To the scientist, the universe is a toy box full new toys and gadgets to played with and disassembled just to see how they work.

- anonymous

A Word On Greeks, Sports, Debate, etc. Extracurricular activities such as sororities, fraternities, athletics, drama and other artistic endeavors, etc. are important parts of your total education at Pepperdine. However, these activities require a very significant time commitment. **It is your responsibility to keep up in class while involved in extracurricular activities.**

PLAGIARISM AND CHEATING Fortunately, in all likelihood, no one in this class will be subject this paragraph. Plagiarism and cheating are professionally and ethically wrong. There exists a fundamental difference between working cooperatively (e.g. working together with friends or in a study group on homework problems which this instructor not only approves of but also recommends) and simply copying someone else's work. Cheating on an exam or plagiarizing the work of others is an offense of considerable magnitude. Students suspected of cheating or plagiarism will be

referred to the University Academic Ethics Committee. *It's not worth it – trust me on this!*

**CELLULAR
TELEPHONES,
and INTERNET
MESSAGING**

If you bring a cell phone with you to class, please turn it off or deactivate any audible signals before class starts. It is very distracting (and stunningly inconsiderate) to have incoming calls during class time. Some like to use their computer to take notes in class – a practice which your professor does not discourage. There is a strong temptation to interact through social media during class – a practice which your professor strongly discourages. ***Your course grade will be adversely affected if your phone audibly rings or if you are found to be texting or using social media during class.***

One purpose of a liberal arts education is to make your head a more interesting place to live inside of for the rest of your life.
- President McPherson, Bryn Mawr College

**OTHER PET
PEEVES**

There are really very few things that bother this professor during class time. An open and friendly classroom that allows for discussion and dialog is desired and, even, encouraged. However, there are a few behaviors that can elicit a strong and negative response. The chances of getting along with this and other professors are greatly increased if you avoid...

- ...continued chatter past the scheduled start of class time.
- ...talking when the instructor is talking or another student is asking a question or speaking.
- ...habitually arriving late to class.
- ...chronically leaving and returning to the classroom during lectures.
- ...making any noise while chewing gum.
- ...eating loud food or slurping through a straw during lectures.
- ...failing to laugh at your professor's jokes regardless of their humorous quality.

**IMPORTANT
INFORMATION
THAT DOESN'T FIT
ELSEWHERE**

The incomplete grade (I) will be assigned only in cases of an extreme emergencies and only in the last 3 weeks of class (after Exam 3 but prior to the final exam). According to university policies, the grade of incomplete will not be assigned to allow extra time for a student improve their grade but, rather, only in the case where an emergency prevents a student from completing a course's culminating assignments and exams. Supporting documentation is required. Should the need arise for non-emergency situations, there are 3 opportunities during the semester to withdraw from this course.

**SAVING GRADED
MATERIAL**

It is your responsibility to save all graded materials (exams, homework, etc.) for this class. As per university policies, all grade disputes must be settled by the midpoint of the next non-summer semester which immediately follows this course.

**COUNSELING
CENTER and
DISABILITY
SERVICES**

Students who feel that they may suffer from "test anxiety" or other academic obstacles despite exercising reasonable study and social habits may benefit by speaking to one of the staff in the Counseling Center.

Any student with a documented disability (physical, learning, or psychological) needing academic accommodations should contact the Disability Services Office (TCC264, x6500) as early in the semester as possible. All discussions will remain

confidential. Visit www.pepperdine.edu/disabilityservices/ for additional information.

COURSE EVALUATIONS At the end of every course, each student has the opportunity to evaluate the course and the professor. This input is valuable for every faculty member so that they can discern both what is being well-presented as well as what may need to be modified to improve the course. Course evaluations are completed on-line near the end of the semester.

Your professor in this class appreciates your critique, both good and bad, and believes that you do not need to be motivated to complete your evaluation by receiving “extra credit” points or other intangible rewards.

Guide to Learning

The following taxonomy¹ summarizes the 6 levels of learning. Generally, it may be said that a student who wishes to master the material of a class will strive to reach level 6.

Notice that to move up in the learning hierarchy, a student will have, for example, mastered the language of the field and possess a knowledge-base of basic facts before they can select the correct formula to solve a problem given a list of data. A level 6 “thinker” will necessarily have mastered the lower levels to such an extent that they can call upon those tools as necessary to solve the problem at hand. It is suggested that one cannot effectively move to higher levels until lower levels have been adequately addressed.

1. **Knowledge** Language of chemistry, nomenclature, facts, memorization
2. **Comprehension** Qualitatively predict outcome of a reaction or process, summarize results, estimate a result
3. **Application** Use formulas to solve a problem ($d=m/v$, $PV=nRT$, etc.), apply and calculate, algebraic manipulation, explain and demonstrate
4. **Analysis** Gather and use experimental data to solve an assigned problem, present results in written or oral format
5. **Synthesis** Use prior knowledge to derive new knowledge, derive from known equations new and useful equations, utilize prior material learned in prior courses in current course, read the primary and secondary literature to obtain necessary tools for performing an experiment, independently design a new experiment or analysis, gather and use experimental data to solve a problem, write and speak clearly and accurately in the scientific style
6. **Evaluation** Examine data and results to distinguish quality from “noise”, read the primary literature and rationally and critically discuss the results presented, predict the outcome of similar experiments

¹This hierarchy is based on Bloom's Taxonomy of Cognitive Learning

Some Important Dates

1/17/2020	Last day of add/drop period
1/20/2020	Martin Luther King holiday
1/28/2020	Last day to change CR/NC status
3/2/2020	Spring Break
3/16/2020	Last day to withdraw with a grade of W
4/17/2020	Last day to withdraw with a grade of WP/WF
4/29/2020	Final exam: 7:30-10:00a

A Note on Grades

In all likelihood, you have been in one or more of the classes taught by this instructor. That being the case, you have seen this grading criteria before. Please take a moment and review it as a reminder of how this course will be graded.

Most students believe their course grade is important, and rightly so. However, many will try to "earn" an 'A' with a minimum amount of work, or with procrastination, cramming, etc., and perhaps even cheating. One goal of this instructor is to help you make the attainment of knowledge (not just chemistry) and its wise use your ambition. When you make learning *personal* and not simply a short-term goal to get you to the next class, job, etc. then the grade will have a new significance and be a by-product of (rather than) the goal. With this in mind, the guidelines for the definition of the course grade is outlined below. The will be the criteria used to determine your course grade

A is the highest academic grade possible. This honor is **not** automatically given to a student who ranks highest in the course, but is reserved for accomplishment that is truly distinctive and demonstrably outstanding. It represents a superior mastery of course material and is a grade that demands a very high degree of understanding, originality, and/or creativity. Further, the student is characterized as one who takes initiative in seeking new knowledge outside the formal confines of the course.

B is a grade that denotes achievement considerably above acceptable standards. Good mastery of course material is evident and student performance demonstrates a high degree of originality, creativity, or both. Student works well independently and often shows initiative. Oral and written analysis, synthesis, and critical expression is considerably above average.

C indicates a satisfactory degree of attainment and is the acceptable standard for proceeding to more advanced work in the field. It is the grade that may be expected of a student of average ability who gives to the work a reasonable amount of time and effort. This grade implies familiarity with the content of the course and acceptable mastery of the material. Student displays some evidence of originality, creativity, or both. Student works independently at an acceptable level and **completes all requirements in the course**, including attendance and participation.

D denotes a limited understanding of the subject, meeting only the minimum requirements for passing the course. It signifies work which in quality and/or quantity falls below the average acceptable standard for the course. Performance is deficient in analysis, synthesis, and critical expression and lacks in originality and creativity. This grade is insufficient to proceed to higher level courses in the discipline. For most students this grade is the result of insufficient devotion of time to the course.

F indicates inadequate or unsatisfactory attainment and a serious deficiency in understanding of material. This grade also indicates the student cannot work independently and/or fails to complete assignments. This grade is usually earned by students who do not attend class or devote sufficient time to study. This grade, like the 'D', is inadequate for proceeding to higher level courses in the field.

Generally Observed Student Characteristics

(Adapted from John H. Williams, *The Teaching Professor*, 1993, pp 1-2)

The "A" Students...

- Attendance** ...have nearly perfect attendance.
- Preparation** ...are prepared for class. Their attention to detail is superb and they usually read the material prior to class.
- Curiosity** ...show a high level of interest in the subject matter whether they actually like the subject or not. They look up or search out answers to topics that they don't understand. They often ask interesting questions or make insightful comments.
- Retention** ...are able to retain new material and consciously connect past learning to the present.
- Attitude** ...have an attitude that displays both the determination and self-discipline required for success. They also show initiative and do things without being told.
- Talent** ...possess a special talent. It may be exceptional intelligence and insight or it may be unusual creativity, organizational skills, commitment and perseverance - or a combination thereof. These gifts are evident to the professor and usually to the other students as well.
- Results** ...make the highest grades on tests and their work is generally a pleasure to grade.

The "C" Students...

- Attendance** ... put other priorities ahead of academic work and may miss class frequently.
- Preparation** ...prepare their assignments consistently, but in a perfunctory manner. Their work may be sloppy or careless and at times is incomplete or late.
- Attitude** ...are not visibly committed to the class. They participate, if at all, without enthusiasm and their body language often expresses boredom.
- Talent** ...vary enormously in talent. Some have exceptional ability, but show undeniable signs of poor self-management or bad attitude. Other are committed and diligent, but are simply average in academic ability.
- Results** ...obtain mediocre or inconsistent results on tests. They have some concept of and familiarity with the material, but clearly do not show mastery of the subject matter while insisting otherwise.

Advanced Analytical Chemistry Contents and Coverage

Module		
1	Nuclear Magnetic Resonance Return to theory Proton, carbon, and other nuclei	Chapter 19 Lab: 1-D/2-D experiments, Nuclear Overhauser Effect, DEPT, COSY
2	Electroanalytical Chemistry Electrochemical cells Electrode potential Current Electrode types Overvoltages and other voltage drops	Chapters 22,23,24,25 Lab: theoretical vs real cell potential, construction of the galvanic cell, voltammetry, cyclic voltammetry of reversible and irreversible systems, redox titration
3	Selected Topic(s) Instrumentation Applications	