Instrumental Analysis Laboratory – Chemistry 341
Spring 2020

See Lecture Syllabus for additional informatino

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Location: KSC 430
Time: Thursday, 2-6 p.m
Text: Green, *Laboratory Manual to Accompany Quantitative Chemistry and Instrumental Analysis*
Other: Scientific calculator, laboratory notebook, safety goggles or glasses, lab coat

PHILOSOPHY

The purpose of laboratory is to provide an opportunity to perform specific sample preparations and chemical analyses of a variety of substances and mixtures using instrumental methods and to provide reinforcement for and examples of the chemical and analytical principles discussed in the lecture.

The overall goal of this course is to provide participants with a sufficient understanding of the principles, laws, and theories of analytical chemistry and instrumentation for chemical analysis to enable them to successfully analyze real samples using selected classical and instrumental methods. The student should gain the competence to follow a standard procedure, operate the equipment 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. Analysis of small samples is emphasized using methods such as spectroscopy, spectrometry, and chromatography. A working knowledge of information obtained from prior courses (including general chemistry, quantitative chemistry, physics, and mathematics) is essential for any level of success in this course.

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 utilize analytical chemical instrumentation properly including: preparation of high accuracy standards, set the operating parameters of different instruments, and perform calibration and analysis.

GOALS

While the major foci are on the principles and capabilities of selected analytical methods and techniques, the specific goals are that every participant will…

- ✓ learn to maintain and use precision electronic analytical equipment properly and appropriately for the analysis at hand.
- ✓ realize the existence of a variety of analytical methods, each with its own particular capabilities and limitations.
- ✓ develop a basic understanding of the electronic, optical, mathematical, and mechanical subsystems in each instrument introduced or used in the laboratory.
- ✓ understand that the selection of one method as being superior to another in the solution of a particular analytical problem, is based on such factors as sensitivity, time required, selectivity, the economics involved, etc. Also, understand that the success of any of the analytical methods used depends upon a working knowledge and control of the chemical reactions, operating parameters, accuracy and precision required, and time available.
- ✓ develop or refine the chemical intuition necessary to know where physical steps in an analysis may be modified such that accuracy and/or precision in the the analysis is not compromised.

- ✓ recognize and appreciate the value of the analytical equipment used, as well as their limitations, in the solution of selected problems faced by the technician.
- ✓ be instilled with quantitative analytical habits and skills that, in spite of possibly never performing a specific chemical analysis from this course again, will be valuable regardless of the scientific field the participant may end up in.

The laboratory course is nearly entirely a course in applied chemical analysis. Because of this characteristic, each participant must come to the laboratory with the foundational skills expected to have been mastered in previous chemistry courses.

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

- ✓ demonstrate the meticulous quantitative analytical skills necessary to perform accurate chemical analysis as well as independently operate each instrument introduced in the laboratory.
- ✓ properly report analytical results with appropriate statistical analysis and confidence.
- ✓ carry out calibration, 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.
- ✓ solve a variety of numerical problems dealing with the analysis of samples using computers and a variety of different software packages.
- ✓ write in scientific format reports of the theory, experimental method, and results of an analysis.

REQUIRED MATERIALS

Investigation monographs will be distributed on a weekly basis, as in Quantitative Chemistry (CHEM 340).

A permanently bound (not spiral or loose-leaf) laboratory notebook; quadruled preferred – all procedures, data, results, calculations, and solutions to problems will be recorded in the laboratory notebook. A lab notebook with yellow removable pages is neither required nor preferred. If there are sufficient pages left in your Chem 340 laboratory notebook, you can simply append experiments, investigations, and analyses to the existing notebook.

Scientific calculator

Approved laboratory safety goggles (available in bookstore or through the SAACS)

Lab coat

ATTENDANCE

You are required to attend prelab discussion and laboratory at the assigned time. The lab is scheduled for 4 hours. You will undoubtedly, at times, take longer than 4 hours. You will have some opportunity to work on your laboratory assignments at other times as well if you are not finished by 6 p.m (but see below, **AFTER HOURS WORK**). This offer of extra time does not extend to those who leave with the intention to come back later to finish – it is only offered to those who actually work during the assign 4 hours and cannot complete the lab on time. I will give time-saving hints when they are available and appropriate (and don't adversely affect the analysis or experiment). **If you miss more than 3 labs, a lab grade of 'F' will be assigned.** Please don't miss lab. **The laboratory grade is independent of the course grade**.

You may not miss lab and make it up later. There will be lecture, discussion, and problem solving sessions during the beginning of each lab. **If you miss any part of 3 lab periods**, you will be assigned a grade of 'F' for the lab and the course.

LABORATORY NOTEBOOK

A laboratory notebook must be kept for every analysis performed. The style of the book and the format of the entries will be described in lab and is also outlined in the laboratory textbook. *The laboratory notebook will be rigorously graded.*

LABORATORY REPORTS

For analyses and experiments which require a formal report, a written report of the results of your laboratory work is due at the beginning of the second laboratory period following the completion of the work unless otherwise informed. Reports turned in within 1 week of the date the analysis was assigned will receive a 10% bonus. It is at the instructor's discretion to cancel the 10% bonus policy. Late work will be assessed a penalty of no less than 10% per day starting the first day after the report is due. The format of the report will be described in lab and is outlined in the laboratory textbook. **The laboratory report must be typed and must be scientifically and grammatically correct. Spelling and sentence structure counts.**

You may turn in laboratory reports electronically as a email attachment, should you desire. Laboratory reports submitted in this manner must be submitted in Microsoft™ Word® format and the entire report must be self-contained in one file. The instructor will **not** open more than one file per lab report.

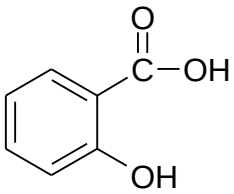
Handwritten chemical formulae, chemical structures, and mathematical equations will be accepted. However, your instructor recommends the following software for Microsoft Windows users:

Chemical Structure Drawing:

Accelrys Draw (Dassault Systemes), ACD/ChemSketch (Advanced Chemistry Development Laboratories), or KnowItAll (KnowItAll Informatics System, BioRad). Knowing the rules of writing structures, you can convert

$C_7H_6O_3$

into



quickly with a very professional appearance.

The best thing about these three chemical drawing packages is that they are free. Links to ISIS/Draw, ACD/ChemSketch, and KnowItAll are available at the Cool Links area of the course website.

Your instructor does not know of any equivalent Mac programs which are free.

Chemical Formula Formatting:

Christopher King's *Chemistry Formatter* add-in for MS Word and MS Excel is an excellent macro add-in if you use the Microsoft Office suite. There are no versions of the add-in for other word processor or spreadsheet programs. The intelligent chemistry formatter, when properly installed, eliminates the need to select 'format|font|subscript' for the formula stoichiometry in a chemical formula and usually gets the charge placement correct on ions. You can also get excellent looking chemical equations quickly. For example, consider the chemical equation

C6H12O6 + 6 O2 --> 6 CO2 + 6 H2O

Simply highlight the entire equation, apply the chemistry formatter add-in with one mouse click, and the equation becomes

$C_6H_{12}O_6 + 6\ O_2 \rightarrow 6\ CO_2 + 6\ H_2O$

Additionally, showing a number in proper exponential notation is easy. Perhaps, because of textual context, a result such as 0.00115 g needs to be presented in exponential notation. Since typing the value in proper exponential notation syntax is time consuming, there is a

temptation to simply type 1.115E-3 since, afterall, that's the way the value is displayed on the calculator. However, with the *Chemistry Formatter*, the unconventional (and incorrect) presentation shown previously is easy to correct. By highlighting the value and applying the formatter, 1.115E-3 g in one mouse click becomes 1.115×10^{-3} g.

King's *Chemistry Formatter* is free to download and use. A link to the *Chemistry Formatter* can be found in the Cool Links area of the course website.

Equation Writing and Editing:

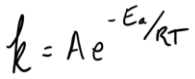
The Microsoft Office suite comes packaged with a good equation editor (aptly named *Equation Editor* but it is not activated during a normal installation of the suite. Simply run the install CD, customize the installation, and add the Equation Editor.

A better equation editor is *MathType* (Design Science, Inc.) for Windows and Mac. It is actually the full version of MS *Equation Editor* and is extremely powerful.

Using the MathType add-in, you can easily make

$$k = A \exp[-E_a/RT]$$

or the handwritten



look like a typeset equation:

$$k = A \cdot e^{-\left(\frac{E_a}{RT}\right)}$$

Equation Editor is included with Microsoft Office Suite if you have the installation CD. *MathType* can be purchased with an academic discount directly from Design Science, Inc.

LABORATORY EXAMS

There are no laboratory exams scheduled although information learned in the lab will be found on lecture exams. You must treat laboratory as seriously as the lecture. It is at the discretion of the instructor to give unannounced pre-lab quizzes if situations warrant.

AFTER HOURS WORK

There will be some opportunity to work outside the normal confines of the scheduled lab time. This privilege is to provide you sufficient time to perform excellent quality work under a minimum of time pressure. This does NOT mean that you can work on an analysis at your leisure. The opportunity to work on laboratory analyses at unscheduled times is a privilege. This privilege can be revoked individually or for the entire class. **This level of freedom in the laboratory <u>will</u> be revoked immediately for anyone who works without protective eyewear. This is a "zero-tolerance" policy and is not negotiable.** Final word... it is highly recommended that you work with someone in case of emergency.

You may contact the professor by e-mail at any time and by phone in emergencies (be very loose with your definition of an emergency).

COMPUTERS

There is access to computers in the KSC laboratories. Computers and printers in the laboratories are not for printing notes, handouts, reports, etc. for any class. Violation of this policy will result a complete prohibition on using the computers.

Under no circumstances install software on any of these computers without consent of the instructor. If you use the computers to access email, don't forget to log off.

GRADING

Your overall lab score will be calculated approximately evenly between all laboratory reports and lab notebook entries. Laboratory attendance, efficiency and timeliness in completing analyses also contribute significantly to your lab grade. Your lab grade will be based on the same fixed scale as lecture.

Approximately 4% of the laboratory grade is based on lab cleanliness and safety. These are not "earned" points: points will be deducted at the end of the semester for violations. The following list will give a representative guideline of how the grade is assessed.

- Reagent bottles must be kept closed except during transfers.
- Balance areas must be kept clean at all times and spills on the balance pan and inside of the windscreen must be remediated immediately.
- At the completion of an investigation or analysis, glassware will be cleaned and placed back on appropriate shelves or in cabinets and drawers.
- Spills on hood decks and benchtops must be cleaned up before leaving lab.
- Hotplates and stirplates should be stored in the cabinet as neatly as possible.

Much of lab cleanliness and safety should be self-policed. Those "caught" cleaning up after others will be rewarded. This is a shared laboratory, so courtesy is expected for the next users.

A Word On Sorority, Fraternity, Sports, *SongFest*, 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. As a scientist you have certain ethical responsibilities with respect to data collection, recording, and analysis. Data that has been collected in the laboratory can never be changed to suit your expected outcomes. Plagiarism and cheating are professionally and ethically wrong. There seems to be a strong temptation for the students in the scientific community to plagiarize material so as to improve their course grades. 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. Plagiarizing the work of others is an offense of considerable magnitude. For assignments in which collaboration with your peers and other faculty is considered acceptable, you are encouraged – perhaps even expected – to do so provided you include a list of collaborators when submitting your assignments.

You are expected to conduct yourselves per the terms of the Seaver College Code of Academic Ethics. Any cheating (including plagiarism) will be punished as severely as allowed under University guidelines. Please see the laboratory instructor or the Seaver College Student Handbook for any questions about this policy. Students suspected of plagiarism will be assigned a grade of zero for that work and may be referred to the University Academic Ethics Committee. *It's not worth it – trust me on this!*

SAFETY IN THE LABORATORY

Safety Goggles

Safety goggles or safety glasses (meeting ANSI Z87.1 standards or greater) must be worn in the laboratory at all times when any chemical procedures are underway. Safety eyewear may be purchased at the bookstore or through SAACS. Any student who is not wearing safety eyewear will be asked to leave the laboratory, will not be allowed to make up the laboratory, and will receive a grade of zero for that investigation. The use of safety eyewear in the laboratory is a zero-tolerance policy and is governed by university regulations and local, state, and federal laws.

Appropriate Dress

Students must dress appropriately for laboratory work. This means wearing a lab coat at all times. Open-toed shoes and sandals are unacceptable in lab (this includes clogs and crocs). **You will be asked to leave and change clothes or shoes, if needed.** Make sure you come prepared, especially if you live off campus. Appropriate laboratory attire is a zero-tolerance policy and is governed by university regulations and local, state, and federal laws.

Hair

If your hair is longer than shoulder length prudent laboratory practices suggest it should tied behind your head in order to avoid accidental contact with open flames or chemicals that might be on the lab bench.

Electronic Equipment

Audiovisual or electronic equipment (except for calculators and computers for data collection and analysis) are not allowed in the laboratory without permission. Permission is given freely, but restrictions apply.

Food and Beverages

You may not eat, drink, or bring food in the laboratory.

SAVING GRADED MATERIALS

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.

CELL PHONES

If you bring a cellular phone to lab, please TURN IT OFF. It is very impolite and unsafe to have incoming calls during lab. Answering calls during lab is not allowed and will adversely affect the lab grade.

WITHDRAW POLICY

Despite the independent nature and course number of the chemistry lecture and lab, except under extreme and unusual circumstances, you may not withdraw from lab and remain in lecture, or *vice versa*.

INTELLECTUAL PROPERTY STATEMENT

Course materials prepared by the instructor, together with the content of all lectures and review sessions presented by the instructor, are the property of the instructor. Video and audio recording of lectures and review sessions without the consent of the instructor is prohibited. Unless explicit permission is obtained from the instructor, recordings of lectures and review sessions may not be modified and must not be transferred or transmitted to any other person. Electronic devices other than calculators (e.g., laptops, cell phones, PDAs, calculators, and recording devices) are not to be used during lectures or exams without prior permission of the instructor.

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.

Disclosure Statement Required by the State of California

Warning: Natural Science's laboratories contain and certain class experiments or procedures which will expose you to chemicals known to the state of California to cause cancer, birth defects, and other reproductive harm at levels which require a warning. For more information, contact your instructor or the Office of Regulatory Affairs at extension 4702.

Instrumental Analysis Laboratory – Chemistry 341
Spring 2020
Weekly Schedule

This schedule is subject to change

Week	Date	Activity
1	Jan 16	Readings: **Chapter 6,13,14 (SHN)** Activity: ***Introduction to Emission and Absorption Spectroscopy: Line spectra, continuous spectra, spectroscopes, spectrophotometers***
2	Jan 23	Readings: **Chapter 6,13,14 (SHN)** Analysis: Electronic Spectroscopy I ***Determination of the Formula of a Complex by the Method of Continuous Variations*** Additional Investigation: ***Determination of Formula and K_f of the Cu-en complex: Does Job's method work?***
3	Jan 30	Readings: **Chapter 6,13,14 (SHN)** Analysis: Electronic Spectroscopy II ***Spectrophotometric Analysis of a Complex Mixture*** Additional Investigation: ***Simultaneous Determination of Aspirin, Acetaminophen, & Caffeine: Is it Feasible?***
4	Feb 6	Readings: **TBA** **A Separation Science Interlude** Analysis: ***Determination of Simple Sugars by HPLC-ELSD After Desalting by SPE***
5	Feb 13	Readings: **TBA** Analysis: **TBA**
6	Feb 20	Readings: **Chapter 15 (SHN)** Analysis: Fluorescence Spectroscopy ***Determination of the CMC of Sodium Dodecyl Sulfate Using a Fluorescent Probe*** Additional Investigation: ***Comparison of Two Instruments for Spectra, Detection limit, Signal-to-Noise, and Results of the CMC determination***
7	Feb 27	Readings: **TBA** **A Second Separation Science Interlude** Analysis: **Gas Chromatography-Mass Spectrometry**
8	Mar 5	**SPRING BREAK**
9	Mar 12	Readings: **Chapter 7-9 (SHN)** Analysis: Flame Atomic Absorption Spectroscopy and Introduction to Automation ***Determination of Ni by standard additions***
10	Mar 19	Analysis: Graphite Furnace Atomic Spectroscopy ***Determination of Cu by direct calibration***

11	Mar 26	Readings: **TBA** Analysis: **TBA**
12	Apr 2	Readings: **Chapter 16,17 (SHN)** Analysis: Vibrational Spectroscopy ***Fourier Transform Infrared Spectroscopy – FT-IR***
13	Apr 9	Readings: **Chapter 19 (SHN)** Analysis: Nuclear Magnetic Resonance Spectrometry I: Proton NMR
14	Apr 16	Readings: **Chapter 19 (SHN)** Analysis: Nuclear Magnetic Resonance Spectrometry II: Carbon NMR
15	Apr 23	Readings: **TBA** Analysis: **TBA**
16	Apr 27	Finals Week

You are allowed to one (1) drawer for personal storage. You may store your books, lab coat, samples for analysis, or whatever, but you may have only one (1) drawer. There will also be a shared cabinet for desiccators and other larger equipment for your use.

You must practice good housekeeping in the laboratory since there will be many users in the laboratory besides yourselves. The lab instructor and/or TA and/or Stockroom Staff will "clean up" unlabeled, unattended, or otherwise messy areas (you have been warned). Proper labeling and storage techniques will be observed.

Replacement "unknowns" will cost 10% of the report score for that analysis unless otherwise notified.