

CHEM 4461/CHEM 5461
INSTRUMENTAL ANALYSIS (Spring 2013)
LECTURE COURSE SYLLABUS

Instructor: Dr. Kevin A. Schug
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Office hours: W noon – 1 pm, F 11 am – noon, stop-in, or by appointment

Text (required): Christian, Dasgupta & Schug *Analytical Chemistry*, 7th Ed.

Additional recommended texts to refer to are Skoog, *Instrumental Analysis* and Harris, *Quantitative Analysis*

<u>Class Schedule:</u>	Lecture:	SH 105	Section 001: M, W 11 – 11:50 am
	Lab:	CPB 215	Section 101: M, W 1 – 4:50 pm
		CPB 215	Section 102: T, Th 1 – 4:50 pm

<u>Grading:</u>	Exams/Homework	400 (4 x 100 pts.)
	Final	200
	Laboratory	400 (4 x 100 pts.)

Description and Goals of the Course: This course explores the fundamental basis of chemical analysis. It is designed to give the student a solid conceptual ground to understand how a given analytical technique works; including its limits and advantages. The emphasis is on solutions analysis and the course is roughly divided into: (i) Basic measurements and concepts; (ii) spectroscopy; (iii) chromatography; and (iv) mass spectrometry.

Tests, Finals, and Grading: At least a 10-point grading scale will be assumed (e.g. 90-100 A; 80-89 B; etc.), however the instructor reserves the right to adjust this scale to accommodate the spread of grades in the course. All exams will be given on the date specified by the instructor (approximate dates are given at the end of the syllabus). No make-up exams will be given. Students scoring an average of 90% or greater on all four regular exams will receive an “A” and are exempt from the final exam. The comprehensive final exam will be given on the date assigned by the University. All exams (except for the comprehensive final)

will be worth 90 points. The remaining 10 points of exam score will be calculated based on homework (see below).

Homework: We will be using Sapling Learning, an on-line homework program, for homework this semester. Use of the system will cost you approximately \$30 for the semester. Below are directions on how to access Sapling Learning and set up your account.

1. Go to <http://saplinglearning.com> and click on your country ("US Higher Ed" or "Canada") at the top right.
- 2a. If you already have a Sapling Learning account, log in and skip to step 3.
- 2b. If you have a Facebook account, you can use it to quickly create a Sapling Learning account. Click "Create an Account", then "Create my account through Facebook". You will be prompted to log into Facebook if you aren't already. Choose a username and password, then click "Link Account". You can then skip to step 3.
- 2c. Otherwise, click "Create an Account". Supply the requested information and click "Create My Account". Check your email (and spam filter) for a message from Sapling Learning and click on the link provided in that email.
3. Find your course in the list (you may need to expand the subject and term categories) and click the link.
4. Select a payment option and following the remaining instructions.

Once you have registered and enrolled, you can log in at any time to complete or review your homework assignments. During sign up or throughout the term, if you have any technical problems or grading issues, send an email to support@saplinglearning.com explaining the issue. The Sapling Learning support team is almost always faster and better able to resolve issues than your instructor.

Several sets of homework will be assigned between each test period. All homework should be completed by the set due dates. The average of the grade earned on all homework sets within a testing period will form the basis for up to 10 points toward each exam score (i.e., if the average grade earned on all assigned homework between Exam 1 and Exam 2 is 83%, then you will be awarded 8 out of 10 possible homework points on Exam 2). Ample time is given to complete all homework sets according to the availability of your schedule, but you are advised not to leave completion of homework assignments until the last minute.

Tools for Success in Lecture:

1. Attend class!! Complete assigned on-line assignments
2. Keep up with material. Review expected learning outcomes (below) and read relevant chapters before lecture. Formulate questions if a concept is unclear.
3. Consult multiple texts.
4. Dedicate appropriate study time. In Chemistry, you should consider spending three (3) hours studying outside of class for every one (1) hour of lecture.
5. Review your lecture notes after every class and seek to clarify any points which are unclear.
6. Complete all of the homework problems in a timely manner; ideally, following each lecture.
7. Don't procrastinate. These concepts take time and practice to sink in, so do not leave studying until the night before an exam.
8. Form a study group. Meet regularly to solve problems together and obtain help with difficult concepts. Collect contact info for each of your study group members.

Student Learning Outcomes (lecture by lecture)

The in-person course content will be supported with on-line content from ChromACADEMY (www.chromacademy.com). A free registration is given for all University students for a period of five years (you should use your mavs.uta.edu email account, because usage of the site will be tracked). While specific tutorials, webcasts, and assessments are prescribed below, you are encouraged to more fully explore the resources on this exceptional website.

Lecture 1.1 (Test 1) (1/13/2014): Students can convey the basic role of an analytical chemist and what steps are associated with designing and performing analytical analyses.

Lecture 1.2 (Test 1) (1/15/2014): Students can apply appropriate basic statistical concepts and tests for the treatment of analytical data predominantly subject to random error. Appropriate conclusions can be drawn based on the results of statistical analysis.

Lecture 1.3 (Test 1) (1/22/14): Students can appropriately choose and apply different quantitative calibration and analysis techniques (external standard,

internal standard, standard addition) for determination of unknown concentrations. Students can relate the advantages and disadvantages of each of these techniques.

Lecture 1.4 (Test 1) (1/27/14): Students can relate the performance of a method through the determination of commonly prescribed method validation parameters (accuracy, precision, linearity, limit of detection/quantification, robustness, etc.).

Lecture 2.1 (Test 2) (1/29/14): Students can differentiate and describe the different ways (diffraction, refraction, reflection, polarization, absorption) that light can interact with matter. They can also describe how a change in the energy of light changes how light is absorbed by different chemical species.

Lecture 2.2 (Test 2) (2/3/14): Students will correlate infrared spectroscopy and the energies associated with IR light with the vibrational excitation of different functional groups of molecules. Students will correlate ultraviolet/visible spectroscopy to electronic excitation of chromophores in a molecule. They will be able to convey the advantages and limitations of Beer's Law for correlating UV/Vis absorption with analyte concentration.

Lecture 2.3 (Test 2) (2/5/14): Students can convey detailed aspects of the different instrumental hardware components that make-up photometers and spectrophotometers. Where multiple choices exist for a given component (e.g. detectors), students can draw a representation of each and describe the differences, advantages, and limitations of each.

Lecture 2.4 (Test 2) (2/12/14): Students can convey terms and time-frames associated with absorption, relaxation, and emission events in the form of a Jablonski diagram. Students can describe what chemical functional units and solution conditions favor and disfavor (including quenching) fluorescence and phosphorescence processes.

Lectures 2.5 (2/17/14) and 2.6 (2/19/14) (Test 2): Students can describe hardware needs for atomization (and excitation for emission) experiments. They can convey differences between atomic absorption and emission spectroscopy including how temperature modulates populations of excited and ground state atoms, and ultimately sensitivity of these different techniques for different atoms. Students can provide advantages and limitations for flame, graphite furnace, and inductively-coupled plasma source instruments.

Lecture 3.1 (2/24/14) (Test 3): Students can describe how chemical compounds partition between two separate phases in a liquid-liquid extraction (LLE) sample preparation. They can also describe how different functional units control the hydrophobicity of different chemical compounds, and how this is described for neutral ($\log P$) and ionizable ($\log D$) species. Students can calculate percent extraction efficiencies given various values for a LLE system.

Lecture 3.2 (2/26/14) (Test 3): Students can describe the set-up, components, and steps associated with a solid phase extraction (SPE) experiment. They can connect modes of separation with concepts of phase partitioning and chromatography. They can also contrast SPE with solid phase microextraction (SPME), and they can describe advantages, limitations, and general operational consideration of SPME.

Lesson 3.3 (3/19/14) (Test 3): Students can describe general chromatographic concepts and define pertinent terms. They can perform basic calculations associated with common chromatographic parameters (capacity factor, theoretical plates, resolution, etc.). Students can convey how various factors, including flow rate, control the efficiency of separations vis-à-vis the Van Deemter equation.

Lesson 3.4 (3/24/14) (Test 3): Students can describe the different components of a gas chromatograph and some differences in how these components function for various applications and analytical needs.

Lesson 3.5 (3/26/14) (Test 3): Students can describe the different components of a liquid chromatograph and some differences in how these components function for various applications and analytical needs.

Lesson 3.6 (3/31/14) (Test 3): Students can recount other less main-stream techniques that can be used to separate chemical compounds and their details, potentially including capillary electrophoresis, countercurrent chromatography, etc.

Lesson 4.1 (4/7/14) (Test 4): Students can describe general mass spectrometry concepts, such as accurate mass, resolution, mass accuracy, etc. They can also use basic physics and mathematical concepts to describe how an ionic particle interacts with electric and magnetic fields.

Lesson 4.2 (4/9/14) (Test 4): Students can describe how ions are manipulated in the context of different mass analyzers used in mass spectrometry instruments. They can compare and contrast the advantages, disadvantages, operational aspects, and applications of different mass analyzers.

Lesson 4.3 (4/14/14) (Test 4): Students can describe historical and contemporary means for atomic mass spectrometry, including important concepts (source hardware, interferences, applications, performance) associated with each.

Lesson 4.4 (4/16/14) and 4.5 (4/21/14) (Test 4): Students can describe contemporary means for performing molecular mass spectrometry, including important concepts (source hardware, applications, performance) associated with each. Detailed knowledge related to comparing and contrasting mechanisms and applications associated with different molecular ion sources (e.g., electron ionization, electrospray ionization mechanism, MALDI) has been assimilated.

Lecture Schedule and On-line Support Material:

Date (Day)	Material/Activity	Book Ch.(s)	Test
1/13/14 (M)	Intro (syllabus); Role of analytical chemist	1	1
1/15/14 (W)	Statistics and statistical tests	3	1
1/20/14 (M)	NO CLASS (MLK Jr. DAY)	---	---
1/22/14 (W)	Methods of quantification	Var. (*)	1
1/27/14 (M)	Method validation Chromacademy reading and webcast 1) Analytical Method Validation, Part I (reading) 2) Analytical Method Validation, Part II (reading) 3) Chromatographic Method Validation http://www.chromacademy.com/Chromatographic_Method_Validation_Essential_Guide.html?tpm=1_1	4	1
1/29/14 (W)	Interaction between matter and light	16	2
2/3/14 (M)	Molecular absorption spectroscopy	16	2
2/5/14 (W)	Spectroscopy instrumental components	16, 17	2
2/10/14 (M)	TEST 1 (Test 1 HW due 5 pm Fri. 1/31/14)	---	---
2/12/14 (W)	Fluorescence and phosphorescence	16, 17	2
2/17/14 (M)	Atomic absorption and emission 1	16	2
2/19/14 (W)	Atomic absorption and emission 2	16	2
2/24/14 (M)	Sample Preparation 1	18 (*)	3

2/26/14 (W)	Sample preparation 2 Chromacademy: Sample Prep/Webcast & Tutorials/ "Understanding the Mechanisms of Solid Phase Extraction"	18 (*)	3
3/3/14 (M)	TEST 2 (Test 2 HW due 5 pm 2/21/14)	---	---
3/5/14 (W)	NO CLASS (Complete on-line materials)	---	3
3/10/14 (M)	NO CLASS (Spring Break)	---	---
3/12/14 (W)	NO CLASS (Spring Break)	---	---
3/17/14 (M)	NO CLASS (Complete on-line materials)	---	3
3/19/14 (W)	Introduction to chromatography	19	3
3/24/14 (M)	Gas chromatography Chromacademy: GC/Webcast & Tutorials/ 1) "Developing Better GC Methods-a blueprint" 2) "Split/Splitless Detection" 3) "Developing Fast Capillary GC Separations"	20	3
3/26/14 (W)	Liquid chromatography Chromacademy: HPLC/Webcast & Tutorials/ 1) "Developing Better HPLC (MS) Methods" 2) "Core Shell Particles-Present & Future" 3) "Ion Chromatography"	21	3
3/31/14 (M)	Other separations	21	3
4/2/14 (W)	TEST 3 (Test 3 HW due 5 pm Tu 4/1/14)	---	
4/7/14 (M)	Introduction to mass spectrometry	22	4
4/9/14 (W)	Mass analyzers (Bring laptop, tablet, and/or textbook to class)	22	4
4/14/14 (M)	Atomic mass spectrometry	22	4
4/16/14 (W)	Molecular mass spectrometry 1 Chromacademy: Mass Spec/Webcast & Tutorials/ "Electrospray Ionization (ESI) for LC-MS (Part 1)"	22	4
4/21/14 (M)	Molecular mass spectrometry 2 Chromacademy: Mass Spec/Webcast & Tutorials/ "Characterization of Protein Biopharmaceuticals"	22	4
4/23/14 (W)	TEST 4 (Test 4 HW due 5 pm Tu 4/22/14)	---	
4/28/14 (M)	NO CLASS (Review week)	---	
4/30/14 (W)	NO CLASS (Review week)	---	
5/7/13 (W)	FINAL EXAM (11 am – 1:30 pm)	Comp.	

Denotations of (*) in the Book Chapter(s) column of the table indicate that significant material will be given in notes form, which is not covered in the book.