

# Chem 309 - Instrumental Analysis - Fall, 2009

**Host:** Gary Rice  
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**Office:** ISC 1039B (*spend a lot of time in 1031, my research lab, as well*)  
**Office hrs:** M,W, R, F 11-12; T 1-2, (*or whenever, wherever you can find me*)  
**Phone:** 221-2049 (*office*)  
**TEXTS:** Principles of Instrumental Analysis, 6<sup>th</sup> ed., Skoog, Holler, and Crouch; Chemistry 309 Laboratory Manual (both available from the bookstore at phenomenal prices). I will provide additional materials for specific topics as the semester progresses; Zumdahl (your old Gen. Chem. text) may be useful as well to review some concepts

**GRADING:**

Lab Reports	(12)	260 pts
Hour exams	(3)	350 pts
Problem sets	(4)	250 pts
<u>Final Exam</u>	<u>(1)</u>	<u>300 pts</u>

**For a grand total of: 1160 pts** (*I like bizarre totals*)

{*Note: Final grades are based on a floating curve, with the median grade considered to be a "highly" respectable B*}

## TENTATIVE EXAMINATION DATES:

Friday, September 18 <sup>th</sup>	Exam #1 - Electrochemistry ( <i>90 pts</i> )
Friday, October 16 <sup>th</sup>	Exam #2 - Spectroscopy <u>through</u> atomic emission ( <i>140 pts</i> )
Friday, November 13 <sup>th</sup>	Exam #3 - IR, NMR, and MS ( <i>110 pts</i> )
Tuesday, December 15 <sup>th</sup> @ 8:30 a.m.	<b>FINAL EXAM</b> ( <i>300 pts; comprehensible and comprehensive</i> )

**Objectives:** The primary objective of this course is to develop the tools and knowledge necessary to decide exactly what instrumental techniques will give you the most accurate and useful qualitative and/or quantitative information for a particular analytical problem. A quick scan of the topics on the remaining pages of the syllabus should quickly make you aware of the wide variety of instrumental methods available to scientists, each with its own advantages and disadvantages. We will necessarily have to go at a fairly rapid pace just to become familiar with the basics of each technique, but I want to make this an enjoyable learning process, a particularly challenging goal given the large amount of descriptive material involved. I've always made a point of having a good time while teaching, and certainly don't plan to change.

**Course Outline/Strategy:** The course schedule provided on the following pages contains a fairly detailed outline of the topics to be covered this semester with suggested readings from the text given in *parentheses*. Laboratory exercises related to specific topics are provided as well. I will provide practice problems for most topics during the course of the semester when appropriate. Note that this is only a tentative schedule and only reflects what I think we should cover, not what we will necessarily be able to complete. A copious number of handouts will be provided for topics not sufficiently covered by the text or what I consider to be a better "abstraction of text material". Bottom line is that the class notes and handouts are the key to success completion this course, and the text serves primarily as a reference source for enhancing your understanding of the material. Answer keys for practice problems, problem sets, and tests will be provided to you in a timely manner.

**Hour Exams and Problem Sets:** Four problem sets will be given over the course of the semester. You may use the textbook and class notes for guidance in solving these problems, but absolutely no help or guidance from other individuals except for the first problem set. Hour exams will be given during normal class times on the tentative dates given above. These will be closed book/note exams (other than information that I supply).

## **“TENTATIVE” SCHEDULE**

- Wed 8/26      *Welcome back! Class and lab expectations with seasonal preparations (can you say classical analytical chemistry)*  
— Concentration terms (*Handout; Zumdahl, Pgs 140-46*)  
— Precipitation and Gravimetric analysis (*Handout; Zumdahl, Pgs 751-64*)  
— Buffer solutions (*Handout; Zumdahl, Pgs 716-29*)  
— Titrations: Acid/base (*Handout; Zumdahl, Pgs 744-51*)  
                Precipitation (*Handout*)  
                Complexometric (*Handout*)  
*Practice Problems: (Handout)*
- Fri 8/28-  
Wed 9/2      *What a shocker. Introduction to electrochemistry*  
— Review of basic concepts  
— Galvanic cells, standard potentials, and the Nernst equation  
— Relation of cell potentials to equilibrium, free energy, and formal potentials (*Handout; Recommended Reading: Handout; Zumdahl: Pgs 164-79, 828-37, 841-44; Skoog: Chapter 22: Sections A1-A5, A8, B1, C1-5, C7-8, D, E1-2 Practice Problems: Skoog, Chapter 22: 1, 5ab, 7,8,10,12*)
- Weds 9/2      *Help session for Problem Set #1 (Time and place to be announced)*
- Fri 9/4      *Problem Set #1 due (60 pts)*
- Fri 9/4-  
Mon 9/7      *Now lets turn these spontaneous electrons into analytical tools*  
— Reference and indicator electrodes  
— Glass electrodes and other ion selective electrodes  
— Redox titrations  
*Recommended Reading: Handouts; IA lab manual, Expt #3; Skoog: Chapter 23, Sections A, B, C-1,2,4, D,F1,H5, I Practice Problems: Chapter 23: 5, 6, 10, 13a,c, 14a, 18, 22*
- Wed 9/9-  
Fri 9/11      *Working redox reactions to their full “potential”*  
— Electrolysis and electrogravimetric analysis  
— Coulometry, amperometry, and voltametry (*Harris, Pgs 356-66; IA lab manual; Expt #2*)  
*Recommended Reading: IA lab manual, Expts #1 and 2; Zumdahl: Pgs 855-57, 841-44; Skoog: Chapter 24, Sections A-2,3; B, B1, D, D1, D2 Practice Problems: Chapter 24: 2, 5, 6, 7, 10*
- Mon 9/13-  
Mon 9/21      *And then there was light...or the world of spectroscopy*  
— The properties of light and it's interactions with matter  
— Beer's law and the aberrations that lie within  
*Recommended Reading: Handout; Zumdahl: Pgs 290-97; Skoog: Chapter 6, Sections A, B1-3,5-11, C1-6, D1-2 ; Chapter 13: Sections A, B, C Practice Problems: Chapter 6: 3, 4, 6-8, 12, 18, 19*
- Thu 9/17      *7 p.m. Help session for Exam #1*
- Fri 9/18      *Exam #1 - Electrochemistry (90 pts)*

- Wed 9/23      ***The Legos (building blocks) of spectroscopic instrumentation***  
 Fri 9/25      \_\_\_ Light sources  
                  \_\_\_ Monochromators  
                  \_\_\_ Light detectors  
                  ***Recommended Reading: Handout; Skoog: Chapter 7; Sections A, B,C, D, E, I***  
                  ***Practice Problems: Chapter 7: 1, 11, 12, 14, 22***
- Fri 9/25      *Problem Set #2 due (60 pts)***
- Mon 9/28-      ***Putting applicable spectroscopic instrumentation all together or "it's all in the name"***  
 Wed 10/14      \_\_\_ Atomic absorption and emission spectrophotometry  
                  ***Recommended Reading: Handouts, IA lab manual Expts #5, and Skoog: Chapter 8: Sections A, C1-2; Chapter 9: Sections A1-2, B, C, D1,4-6; Chapter 10: Sections A1,3,4; Chapter 11, Sections A, B, C***  
                  ***Practice Problems: Chapter 8: 1, 5, 6, 9a; Chapter 9: 3, 5, 9, 13a, 16; Chapter 10: 2, 6, 8, 9; Chapter 11: 2, 6, 7***  
                  \_\_\_ UV/VIS molecular absorption spectrophotometry  
                  ***Recommended Reading: Handouts, IA lab manual Expt #4, and Skoog: Chapter 13, Section D; Chapter 14, Sections B, C, D1-2, E***  
                  ***Practice Problems: Chapter 13: 8, 9, 16; Chapter 14: 1, 3, 4, 9***  
                  \_\_\_ Molecular fluorescence spectrophotometry  
                  ***Recommended Reading: Handouts, IA lab manual Expt #6, and Skoog: Chapter 15, Sections A, B, C***  
                  ***Practice Problems: Chapter 15: 1, 2 a-l, 3, 9,***  
                  \_\_\_ Infrared spectrophotometry  
                  ***Recommended Reading: Handouts, IA lab manual Expt #8, and Skoog: Chapter 16: Sections A, B, C; Chapter 17: Section A***  
                  ***Practice Problems: Handouts; Chapter 16: 1, 3, 8, 10a; Chapter 17: 1ab, 2, 4, 5***
- Thu 10/15      7 p.m. *Help session for Exam #2***
- Fri 10/16      *Exam #2 - Spectroscopy through molecular fluorescence (140 pts)***
- Mon 10/19-      ***The organic tool of the trade: Nuclear magnetic resonance spectrometry***  
 Mon 10/26      \_\_\_ Theory of NMR processes  
                  \_\_\_ NMR instrumentation  
                  \_\_\_ NMR other than  $^1\text{H}$   
                  \_\_\_ NMR spectral interpretation  
                  ***Recommended Reading: Handouts, IA lab manual Expt #8, and Skoog: Chapter 19: Sections A1-2, 4, B1-3, C, D, E***  
                  ***Practice Problems: Handouts; Chapter 19: 3, 4, 6, 7ab, 9, 15, 25, 26, 28, 32, 34, 37***
- Fri 10/22      *Problem Set #3 due (70 pts)***

Wed 10/28- ***Molecular Mass Spectrometry: The best of the rest***  
Wed 11/4 \_\_\_ Building a mass spectrometer  
\_\_\_ Ion formation and separation processes in mass spectrometry  
\_\_\_ Mass spectral interpretation  
\_\_\_ Other MS applications  
***Recommended Reading: Handouts; IA lab manual Expt #8, and Skoog: Chapter 20: Sections A, B, C1-3, D, E***  
***Practice Problems: Handouts; Chapter 20: 2, 5, 9, 11ab, 12 ab, 18ab***

**Thu 11/5 7 p.m. *Optional help session on structural elucidation***

Fri 11/6 ***Putting it all together. Structural elucidation using IR, NMR, and MS*** (Handouts, IA lab manual, Expt. #8)

Mon 11/9- ***Separation anxiety: The world of chromatography***  
Fri 11/20 \_\_\_ Theory of chromatographic processes  
\_\_\_ Plate and rate theory  
***Recommended Reading: IA lab manual Expts #9, 10; Skoog: Chapter 26: Sections A-F***  
***Practice Problems: Chapter 26: 3, 5, 6, 14, 16, 17, 22***

**Thu 11/12 7 p.m. *Help session for Exam #3***

**Fri 11/13 *Exam #3 - IR, NMR, and MS (110 pts)***

Mon 11/23- ***So many chromatographies, so little time left***  
Fri 12/4 \_\_\_ Gas chromatography instrumentation and applications  
***Recommended Reading: Handouts and Skoog: Chapter 27: Sections A-F***  
***Practice Problems: Chapter 27: 1, 3, 8, 14, 18, 21, 23, 27***  
\_\_\_ Liquid chromatography  
\_\_\_ High performance liquid chromatography  
\_\_\_ Ion exchange chromatography  
\_\_\_ Size exclusion chromatography  
***Recommended Reading: Handouts and Skoog: Chapter 28: Sections A-G***  
***Practice Problems: Chapter 28: 5, 6, 7ab,de, 10, 11, 14***  
\_\_\_ Capillary electrophoresis chromatography  
***Recommended Reading: Handouts and Skoog: Chapter 30: Sections A – C2***  
***Practice Problems: Chapter 30: 1, 2, 3***

**Wed 12/2 *Problem Set #4 due (60 pts)***

**Fri 12/4 All labs and lab notebook due**

**Tues 12/15 *FINAL EXAM (300 pts) {comprehensive, with approximately 50- 60% on chromatography}***

**THE END**

