

Chem 309 - Instrumental Analysis - Fall, 2011

Host: Gary Rice
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TEXTS: Chemical Analysis, 2nd ed., Rouessac and Rouessac; (available from the bookstore at a reasonable price). I will provide additional materials for specific topics as the semester progresses; Whatever general chemistry text you have may be useful as well to review some concepts.

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| GRADING: | Hour exams (3) | 400 pts | <i>{Note: Final grades are based on a floating curve, with the <u>median</u> grade considered to be a "highly" respectable B}</i> |
| | Problem sets (6) | 160 pts | |
| | <u>Final Exam</u> | <u>240 pts</u> | |
| | For a grand total of: | 800 pts | |

TENTATIVE EXAMINATION DATES:

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| Wednesday, September 21 st | Exam #1 - Electrochemistry (<i>110 pts</i>) |
| Friday, October 22 nd | Exam #2 - Spectroscopy <u>through</u> atomic emission (<i>160 pts</i>) |
| Friday, November 11 th | Exam #3 - IR, NMR, and MS (<i>130 pts</i>) |
| Tuesday, December 7 th @ 9:00 a.m. | FINAL EXAM (240 pts; comprehensive and comprehensible) |

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 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*. 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. The new text is somewhat "shallow" in some areas, thus a number of handouts and supplemental practice problems 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, handouts, and understanding the practice problems are the key for successful completion of this course; 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 through **Blackboard**. I will also communicate periodically through e-mail for any updates, changes, etc.

Hour Exams and Problem Sets: Six "small" 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. **The lowest grade from Problem Sets #2 - 6 will disappear from your class record.** Hour exams will be given during normal class times on the tentative dates given above. These will be closed book/note exams other than the information that I supply, which will be made available prior to the examinations.

“Tentative” Topics, Recommended Readings, and Practice Problems

- Wed 8/24 **Welcome back! Class expectations with seasonal preparations (can you say classical analytical chemistry)**
— **Concentration terms**
— **Precipitation and Gravimetric analysis**
— **Buffer solutions**
— **Titrations: Acid/base, precipitation, and complexometric**
Recommended Reading: Blackboard posting, plus background information on all of these topics may be found in any general chemistry text
Practice Problems: Blackboard Posting
- Wed 8/24-
Wed 8/31 **What a shocker. Introduction to electrochemistry**
— **Review of basic concepts (redox equations, Galvanic cells, standard potentials, and the Nernst equation)**
— **Relation of cell potentials to equilibrium, free energy, and formal potentials**
Recommended Reading: Handouts; Any general chemistry text
Practice Problems: Blackboard Posting
- Fri 9/2-
Mon 9/5 **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; Rouessac – Sections 19.1-19.3
Practice Problems: Blackboard Posting; Rouessac-19.4,19.5
- Wed 9/7-
Fri 9/9 **Working redox reactions to their full “potential”**
— **Electrolysis and electrogravimetric analysis**
— **Coulometry, amperometry, and voltametry**
Recommended Reading: Rouessac – Sections 20.1, 20.9,
Practice Problems: Blackboard Posting; Rouessac-20.6
- Mon 9/12-
Mon 9/19 **And then there was light...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: Handouts
Practice Problems: Blackboard posting; Rouessac-9.1,9.2
- Mon 9/19-
Fri 9/23 **The Legos (building blocks) of spectroscopic instrumentation**
— **Light sources**
— **Monochromators**
— **Light detectors**
Recommended Reading: Handouts
Practice Problems: Blackboard Posting
- Mon 9/27-
Wed 10/13 **Putting applicable spectroscopic instrumentation all together or “it’s all in the name”**
— **UV/VIS molecular absorption spectrophotometry**
Recommended Reading: Handouts, Rouessac-Sections 9.1-9.5, 9.7-9.8
Practice Problems: Blackboard posting; Rouessac-9.4,9.5,9.7
— **Atomic absorption and emission spectrophotometry**
Recommended Reading: Handouts, Rouessac-Sections 13.1-13.9, 14.1-14.3.1, 14.4-14.7
Practice Problems: Blackboard posting; Rouessac - 13.2,13.3,13.4; 14.2,14.6

- **Molecular fluorescence spectrophotometry**
Recommended Reading: Handouts, Rouessac-Sections 11.1-11.3, 11.5-11.6
Practice Problems: Handouts; Blackboard posting; Rouessac - 11.3a,11.4a,b

- **Infrared spectrophotometry**
Recommended Reading: Handouts; Rouessac-Sections 10.1-10.9.2
Practice Problems: Handouts; Blackboard posting; Rouessac - 10.1,10.2,10.3

- Mon 10/18- Mon 10/25** — **The organic tool of the trade: Nuclear magnetic resonance spectrometry**
— **Theory of NMR processes**
— **NMR instrumentation**
— **NMR other than ^1H**
— **NMR spectral interpretation**
Recommended Reading: Handouts; Rouessac-Sections 15.1-15.5, 15.7-15.12, 15.4.1
Practice Problems: Handouts; Blackboard posting; Rouessac - 15.2,15.4,15.7

- Wed 10/27- Wed 11/3** — **Molecular Mass Spectrometry: The best of the rest**
— **Building a mass spectrometer**
— **Ion formation and separation processes in mass spectrometry**
— **Mass spectral interpretation**
— **Other MS applications**
Recommended Reading: Handouts; Rouessac-Sections 3.1-3.6; 3.8-3.11, 3.12
Practice Problems: Handouts; Blackboard posting

- Fri 11/5** — **Putting it all together. Structural elucidation using IR, NMR, and MS (Handouts)**

- Mon 11/8- Fri 11/19** — **Separation anxiety: The world of chromatography**
— **The chromatographic processes**
— **Plate and rate theory**
Recommended Reading: Rouessac-Sections 1.1-1.2,1.4-1.12
Practice Problems: Blackboard posting; Rouessac - 1.12

- Mon 11/22- Fri 12/3** — **So many chromatographies, so little time left.....**
— **Gas chromatography instrumentation and applications**
Recommended Reading: Handouts; Rouessac-Sections 2.1-2.8
Practice Problems: Blackboard posting; Rouessac - 2.1
— **Liquid chromatography and high performance liquid chromatography**
Recommended Reading: Handouts; Rouessac-Sections 3.1-3.6; 3.8-3.11, 3.12
Practice Problems: Blackboard posting; Rouessac - 3.3
— **Ion exchange chromatography**
Recommended Reading: Handouts; Rouessac-Sections 4.1-4.5
Practice Problems: Blackboard posting; Rouessac - 4.1
— **Size exclusion chromatography**
Recommended Reading: Handouts; Rouessac-Sections 7.1-7.5
Practice Problems: Blackboard posting; Rouessac - 7.2
— **Capillary electrophoresis chromatography**
Recommended Reading: Handouts; Rouessac-Sections 8.1-8.3
Practice Problems: Blackboard posting

THE END

Tentative Exam Dates, Problem Set Due Dates, and HELP! Sessions

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|------------------|---|
| Wed 8/31 | <i>Help Session for Problem Set #1 (time and place TBA)</i> |
| Thur 9/1 | <i>Help Session for Problem Set #1 (time and place TBA)</i> |
| Thur 9/1* | <i>Introduction to the Instrumental Analysis Lab (7 p.m. ISC 1127)</i> <i>*Mandatory attendance <u>only</u> for students registered in Chem 309L</i> |
| <u>Fri 9/2</u> | <i>Problem Set #1 due (40 pts)</i> |
| Thur 9/8 | <i>Help Session for Problem Set #2 (7 p.m. ISC 1127)</i> |
| <u>Fri 9/7</u> | <i>Problem Set #2 due (30 pts)</i> |
| Mon 9/19 | <i>Help Session for Exam #1 (time and place TBA)</i> |
| Tue 9/20 | <i>Help Session for Exam #1 (time and place TBA)</i> |
| <u>Wed 9/21</u> | <i>Exam #1 - Electrochemistry (110 pts)</i> |
| Thur 9/29 | <i>Help Session for Problem Set #3 (7 p.m. ISC 1127)</i> |
| <u>Fri 9/30</u> | <i>Problem Set #3 due (30 pts)</i> |
| Thur 10/6 | <i>Help Session for Problem Set #4 (7 p.m. ISC 1127)</i> |
| <u>Fri 10/7</u> | <i>Problem Set #4 due (30 pts)</i> |
| Wed 10/19 | <i>Help Session for Exam #2 (time and place TBA)</i> |
| Thur 10/20 | <i>Help Session for Exam #2 (7 p.m. ISC 1127)</i> |
| <u>Fri 10/21</u> | <i>Exam #2 - Spectroscopy <u>through</u> molecular fluorescence (160 pts)</i> |
| Thur 11/3 | <i>Help Session for Problem Set #5 (7 p.m. ISC 1127)</i> |
| <u>Fri 11/4</u> | <i>Problem Set #5 due (30 pts)</i> |
| Wed 11/9 | <i>Help Session for Exam #3 (time and place TBA)</i> |
| Thur 11/10 | <i>Help Session for Exam #3 (7 p.m. ISC 1127)</i> |
| <u>Fri 11/11</u> | <i>Exam #3 - IR, NMR, and MS (130 pts)</i> |
| Mon 11/21 | <i>Problem Set #6 due (30 pts)</i> |
| <u>Tues 12/6</u> | 9 a.m. - FINAL EXAM (270 pts) <i>Comprehensive, with approximately 50% on chromatography. HELP! sessions TBA</i> |

