Syllabus
Chemistry 314A – Laboratory in Advanced Analytical Chemistry
Spring 2006

INSTRUCTOR:

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COURSE WEB PAGE

A Blackboard page **Analytical Chem (CHEM314A.A, CHEM314A.C CHEM314S SP2006)** has been set up for this course.

CLASS MEETINGS:

Classroom

W 8–8:50 Pfahler 109

Lab (2 sections) <Pre-lab lectures in Pfahler 208; Laboratory in Pfahler 301, 306, and 316>

M1:30–4:20 PM
F 1:30–4:20 PM

OFFICE HOURS

W 3:00–4:00 PM
Th 10:00–11:00 AM

We can also meet at other times, if you set up an appointment. I will often be in my office or the laboratory from 8 to 5 each day. You can try to drop in to see me, but I can not guarantee that I can immediately speak with you.

CATALOG DESCRIPTION:

Laboratory work related to advanced techniques of analysis. The operation of common instrumentation, demonstrations of the effects of instrumental settings on acquired data, and application of statistical methods will be emphasized. Prerequisites: Chemistry 112a or 206a, and 313. One hour of lecture and three hours of laboratory per week. *Two semester hours.*

PRIMARY SOURCES:

Skoog, Douglas A.; Holler, F. James; Nieman, Timothy A. *Principles of Instrumental Analysis, 5th ed.*; Saunders College: Philadelphia, 1998. <Recommended>

Class notes (will be provided)

COURSE GOALS:

The six goals for this course are to demonstrate how different instrumental factors affect the acquired signal, to develop the skills used in performing a good analysis (including sample preparation), to use statistical or chemometric analysis techniques in order to obtain more or better information from your measurements, to show how the thought processes and knowledge gained in learning one technique can be used to understand a

new technique, to deepen and solidify the knowledge that you acquired in Chem 313, and to write excellent notebook entries and reports.

OUTCOMES

You will…

- write a complete laboratory notebook;
- write research papers, which are formatted in the style of a research article in *Analytical Chemistry*, so that you can develop skills to effectively write and read the chemical literature;
- learn how to orally defend your results;
- learn how to efficiently determine experimental conditions for an analysis;
- gain experience in simple troubleshooting of analysis difficulties;
- learn the factors that you need to consider in performing a chemical analysis and how to correctly perform an analysis;
- deepen your knowledge of the techniques to which you will be exposed (GC/MS, AA, UV/Vis, IR, voltammetry, HPLC, and fluorescence) and of basic instrumental theory;
- determine some of the strengths and weaknesses of the various techniques;
- learn how to perform various simple statistical analyses;
- learn how to more completely analyze your raw data; and
- improve your laboratory technique.

COURSE STYLE:

The classroom portion of this course will primarily consist of lectures with some amount of discussion and computer work. The lecture notes will be provided to you, and problem sets will be regularly given to help you develop your skills in the lecture material. The material covered will include statistics, sample preparation, and (additional) information on techniques that were not completely covered in Chem 313.

The lab portion of this course will mainly consist of laboratory work, but some periods will begin with short lectures, which cover practical information not covered in Chem 313 or in the classroom portion of Chem 314A. Five sets of experiments will be performed using seven different techniques: gas chromatography/mass spectrometry (GC/MS), high-performance liquid chromatography (HPLC), infrared spectroscopy (FTIR), UV-visible molecular absorption and fluorescence spectroscopies together, and atomic absorption spectroscopy (AA) and voltammetry together. From these experiments on the individual pieces of instrumentation, you will learn the basic operation of the particular instruments, will investigate how instrumental settings affect the acquired data, will practice statistical and chemometric analysis of data, and will deepen your knowledge of the techniques. During the FTIR rotation students will also participate in a laboratory practical where they will demonstrate their skill in accurately and precisely making solutions. In addition to these experiments, a multi-week, individual and class-wide, unknown project will be performed. In this project each student will be the "expert" on a particular instrument. The class will be given a multi-component unknown mixture, and in addition each student will be given a sample of known identity but unknown concentration. Students will pool their data and assign each other tasks in order to determine the identity of the unknown components (class-wide component), and each student will determine the identity of their unknown (individual component).

You will work in pairs or individually in the laboratory, but all lab reports are individually written. The first five experiments will be performed in two-week rotations. At the beginning of each rotation, you will be provided with the manual for the instrument, with simple instructions on how to operate the instrument, a reference list, and a list of tasks. To help you prepare for the experiment, you must complete a short, prelab

quiz that will be available on Blackboard. During the two-week period you must design experiment(s) for each task that will demonstrate various instrumental and analysis concepts. Work will primarily be performed during the laboratory period, but if necessary and with approval, you may use instrumentation outside of the laboratory period. During the last three weeks the qualitative and quantitative unknown will be performed.

In addition to laboratory manipulations, writing skills will be emphasized. Guidelines will be provided for the writing of the reports and the laboratory notebook. All written reports must follow the format of a research article submitted to *Analytical Chemistry*. The notebook will be kept electronically.

LECTURE OUTLINE

Statistical and Chemometric Analysis
• What is Chemometrics?
• Basic Statistical Terms and Background
• Weighted and Unweighted Linear regression
• Basic statistical tests (t, F, χ^2)
• Hypothesis testing
• ANOVA
• Experimental Design
• Multivariate Analysis
Sampling
(If time) Additional Instrumental Techniques

LAB SCHEDULE

Topics	Assignments Due <Written reports must be received in my e-mail by 7:00 PM on the listed day>
Introduction **Jan. 16, 20**	
Rotation 1* **Jan. 23, 27** **Jan. 30, Feb. 3**	*Quiz must be completed by noon of lab day in first week.* **Report due (Results & Discussion): Feb. 6, 10**
Rotation 2* **Feb. 6, 10** **Feb. 13, 17**	*Quiz must be completed by noon of lab day in first week.* Written Abstract due: Feb. 20, 24 **Oral Results & Discussion: Starting Feb. 20**
Rotation 3* **Feb. 20, 24** **Feb 27, March 3**	*Quiz must be completed by noon of lab day in first week.* **Report due (Experimental and Results & Discussion): March 6, 10**
Rotation 4* **March 6, 10♪** **March 20, 24** **♪ I will not be present on March 10th. Friday lab students will need to reschedule your time to do the lab sometime the week of March 6th.**	*Quiz must be completed by noon of lab day in first week.* **Report due (Introduction and Results & Discussion): March 27, 31**
Rotation 5* **March 27, 31** **April 3, 7**	*Quiz must be completed by noon of lab day in first week.* **Oral Results & Discussion: Starting April 10**
Unknown Analysis **Primarily from April 10 till end of semester**	**Preliminary plans due: April 3, 7** **Final Report and Notebook:** **W: May 3** **F: May 8**
FINAL	**At scheduled lab time or (more likely) TBA during Finals Period**

Notebooks must be e-mailed to me at the end of each lab.

*** Rotation Order: AA:Voltammetry combined, UV-visible fluorescence and absorption combined, FT-IR/Lab Practical, GC/MS, HPLC**

POINT DISTRIBUTION

Total Points 1050

	Points
Final Exam	100
Technique Rotation Reports (Results & Discussion) <Oral or Written>	100 (5x)
Technique Rotations Reports (with Abstract, Introduction, or Experimental)	15 50 25
Final Project Report	150
Rotation Quizzes	5 x 10
Final Project Preliminary Paper	20
Lab Practical	60
Notebook	80

Final Exam

One exam will be taken during the Finals period, covering statistical theory and general laboratory and technique knowledge from the lecture and laboratory. This exam will be scheduled at a time that the whole class can meet during the final exam period.

Problem Sets

Problem sets will be given on the lecture material, but they will *not* be collected. Complete answers will be given. The purpose of these problem sets is to give you practice on some skills that you will use in the laboratory and on which you will be tested in the exam.

Laboratory Reports: Technique Rotations

Each technique rotation report will have a Results & Discussion section. In this section you will be graded upon your results, how well your analysis proves your point, the depth and correctness of your discussion, my observation of how well you use your time and do your experiment in the lab, and your writing (formatting, spelling, sentence structure). This section will generally will be written, although during the second and fifth rotations this information will be presented in a one-on-one oral examination. With one rotation report you will also submit an Abstract, with another an Introduction, and with another an Experimental Section. In these sections you will be graded upon how well written the sections are, the information that you include or exclude, and how well you follow the required format.

Final Report

This will be a complete report with an Abstract, Introduction, Experimental Section, and Results & Discussion. The Introduction and Experimental Section will contain information on the technique that you use during the analysis. The Results & Discussion will contain a report and analysis of the qualitative data that you obtained, the qualitative data from other groups, and the quantitative analysis that you performed. You will be graded upon the quality of your report, your results, and the quality of your analysis.

Rotation Quizzes

Before each rotation, you will need to answer some questions in an on-line quiz to help you prepare for the laboratory.

Final Project Preliminary

Before starting your project, you will outline how you expect to perform your analysis. Prior to the beginning of your analysis, I will sit down with each group and discuss your plan.

Lab Practical

You will be graded on how well you perform procedures used in sample preparation (such as pipeting, using volumetric glassware, etc.). You will be graded on how well you meet certain standards.

Notebook

The notebook will be e-mailed to me at the end of each lab period. You will be graded at random several times during the semester on how well you fulfill the required format, the readability of your notebook, and the information that you include or exclude. The notebook may be supplemented by calculations in Excel or another program and spectra.

Class Attendance and Participation

Although no specific point total is assigned for class attendance and participation, a positive or negative impression in this area can increase or decrease your grade by one step.

Guaranteed Grades

Letter grade	Percentage
Some sort of A	92 %
Some sort of B	82 - 92 %
Some sort of C	72 - 82 %
Some sort of D	62 - 72 %
F	52 - 62%

ACADEMIC POLICIES

Late Policy

Any assignment turned in after the assigned time but before the next day will have 5% of the total points deducted, before the end of one day after the due date will have 10% deducted, and before the end of two days after the due date will have 50% deducted. No assignments will be accepted more than 2 days after the due date, unless special arrangements have been made.

Academic Honesty and Plagiarism

You are responsible for following the rules on academic honesty and plagiarism that are outlined on pp. 13–15 of the *Ursinus College Student Handbook 2005–2006* (a copy can be found on the Ursinus College web page). Because of the ratio of instruments to students, we will work in rotations. Therefore, some students will complete reports and work on a particular technique before other students in the class. To ensure that each student maximizes their own learning experience and is fairly evaluated, students who have previously completed a particular experiment should not discuss with other students how they did a particular analysis or how they presented a particular discussion in their reports, and students who are now working on a particular experiment should not ask other students who are either currently or previously in the class how they did a particular analysis or how they framed an argument in their reports. Any transfer of information of this type will be regarded as an incident of academic dishonesty, and penalties will be assessed. Students can, and are encouraged, to teach other students how to use a particular instrument in a general sense. If you have any questions about what information is fair game, ask me.

Class Attendance

You are responsible for following the rules on class attendance that are outlined on p. 8 of the *Ursinus College Student Handbook 2005–2006* (a copy can be found on the Ursinus College web page). Although I will not formally take attendance in the Wednesday class meetings, your presence, or lack thereof, and participation will be noted and can positively or negatively affect your grade. You are expected to promptly be at all lab periods. If you need to miss or will be late for a particular lab period, you must inform and make arrangements with your lab partner and me prior to the lab period.

EJW Last Revision: 15 January 2006