

Chemistry 346
Instrumental Analysis
Class Syllabus-Spring 2005

Instructor: Dr. Eugene C. Johnson
Office: D-129 Science (346-2888)
Office Hours: 9:00 T, Th (Other times by appointment)
Web Page: <http://www.uwsp.edu/chemistry/ejohnson>
Class Meeting Times:
Lecture: M, W 10:00 SCI A-112
Laboratory: T, Th 11:00 - 1:50 SCI D-114

TEXT: "Principles of Instrumental Analysis, Fifth Edition," by Douglas A. Skoog, F. James Holler and Timothy A. Nieman, Philadelphia: Saunders College Publishing, 1998.

WRITING EMPHASIS: You have the option of enrolling in this course for regular credit or WE credit. The attached WE Assignment sheet outlines the assignments required in addition to those all students in the course will be required to complete. More details about these assignments will be presented at the beginning of class. You must register for the course either for regular credit or WE. You may not switch after the normal drop/add period.

LECTURE: There are two lecture hours per week. During this time we will discuss instrumental methods in a sequence roughly approximating the attached Lecture/Lab schedule. We will discuss as many different instrumental methods as time allows. It will be very beneficial to your understanding of the material if, prior to the discussion of these techniques in lecture, you obtain the information below and come to class prepared to discuss these factors as they relate to each technique:

Name of technique:

Schematic diagram of a typical instrument:

Sources of the analytical signal:

Detectors:

Types of samples:

Sensitivity:

Cost of instrumentation and analysis, including preparation and analysis time:

It is expected that you will come to class having read the assignment from the Lecture/Lab schedule as amended in class. Key questions related to that reading are included with the syllabus. In addition, you will be asked to review the literature for more current information as described later. Lecture time will be used to discuss the readings and clarify points of confusion. We will discuss at the first class meeting how the lecture time can best be used.

When PowerPoint is used in class to present lecture materials, the PowerPoint slides will be available on my web site for your review.

If you are taking this course for Writing Emphasis, WE, credit refer to the WE Assignment Sheet for details on WE assignments.

LABORATORY: The 15 weeks of laboratory will be split between laboratory experiments and preparation, examinations, discussions of the theoretical background of instrumental methods and WE assignments. Lab experiments will be conducted during weeks 3, 4, 6, 7, 8, 9, 11, 12, 13, and 15. During weeks 5, 10 and 14 exams will be administered. In addition, remaining time during weeks 1, 2, 5, 10, and 14 will be used to augment lecture, prepare for the next round of labs, spend time reviewing the literature and discuss WE assignments. Refer to the attached Lecture/Lab handout and WE Assignment Sheet for the weekly activities during the semester.

In lab we will be working in small groups and rotating experiments until each person has completed all experiments. Once the final class roster is established, a lab rotation schedule will be prepared that will specify when and with whom you will work each week. The experiments will be placed into two groups of 5 experiments each. I will do everything possible to familiarize you with the instrumentation and theoretical basis for each experiment, but it is critical that you come to class prepared, having read the background material about the experiment you will be conducting. Because many of you already have some hands-on experience with our instrumentation, I will often ask those of you who have used an instrument to serve as in-class experts to assist others in the proper use of the instrumentation. **After an experiment is completed, the instrument must be returned to its rest state (either turned off, or placed on standby as necessary), and the laboratory area surrounding the instrument cleaned and returned to an orderly condition.** Any problems encountered during the period should be reported to me immediately, and recorded in your laboratory notebook. We will meet as a group at the beginning of each lab period to discuss problems and for me to make announcements. **Attendance at this time is required.** Failure to attend any one of these will result in a decrease in your grade as indicated below.

Laboratory Grading: The laboratory grade will be determined in two parts: (1) Laboratory Notebook and (2) Participation Evaluation.

Laboratory Notebook: The laboratory notebook is the place where all data are collected and recorded, along with observations made during the data collection process. It is these observations that may be ultimately useful in allowing you to successfully reproduce or better yet improve your data. For this reason I recommend that you write on only one side of each page and keep the other side for notations. The laboratory notebook must be turned in to me at the end of class each Monday following the completion of a lab the previous Thursday. I will review it and return it to you Tuesday morning for **15 points per lab.** The WE portion of the grade will be based on the completeness of the data recorded and adherence to the form stipulated below. The audience for this lab book is you and your supervisor. Lab Book Requirements:

1. The lab book should be permanently bound with consecutively numbered pages.
2. The first few pages should be reserved for a table of contents that must be kept up to date.
3. All entries must be legible and well spaced from one another.

4. Enter data directly **in ink** as they are taken. Never recopy numbers or use loose sheets of paper.
5. Cancel errors or rejected data by drawing a single line through them. Do not erase or remove sheets. The notebook should be a permanent, accurate, historical record of the original laboratory work.
6. Each notebook page should be dated and initialed as it is used.
7. The suggested form of the experimental entries is as follows:
 - a. Name of the experiment
 - b. Date experiment was begun (each new page will be dated and signed as it is used)
 - c. Experiment number
 - d. Names of co-workers
 - e. The title of the experiment and reference as to where the full text can be found, for example the lab handout, a notebook, computer file, etc. If the source is not printed, write the experimental details into the lab book.
 - f. Clearly identify any known or unknown samples analyzed along with the number of the unknown, if applicable.
 - g. Reference the procedure used, but record any corrections or modifications that were made in your lab book. If the procedure was part of a handout, you may attach the handout to the book. Attached material should be on the left side of the pages.
 - h. Descriptions of all materials, chemicals, solutions and instruments used in the experiment. For all solutions prepared by you, the exact procedure, including amounts, must be recorded. Use tables where several solutions are prepared similarly. The left side of the page should be used to record observations, for sample calculations, and for showing the calculations used in computing results.
 - i. Enter data on the right side of the page only. Clearly label all entries. To facilitate direct entry of experimental work, it is useful to set up a data page **before** starting the experiment. ALL data collected must be recorded, including weights and volumes used in preparing solutions. Do not presume that you used 1.0 M HCl just because you were told to do so. The lab book should have information on the solutions you actually used. If the collected data are recorded electronically, include the file name for the file and attach a hard copy to the lab book on the left side of the page. All data must be clearly identifiable and dated.
 - j. Instrument used and settings for data collection. Include filename for any stored data.
 - k. At the end of each experiment you should include a results table that lists the best value for the result of the experiment and a statement of the precision attained in the analysis. These results tables can be computer generated and permanently attached to the report if that is the way the data are to be evaluated for the reports. A sample calculation is also helpful in illustrating how the results were obtained. It is always a good idea to include a file name and storage location for data and reports that are saved electronically.
 - k. You should include a brief discussion of your results at the end of each experiment.
 - l. Answers to any questions posed in the experimental instructions for the laboratory experiment.

Participation Evaluation: I will assign each of you up to 5 points per laboratory for your cooperation and participation. Being a productive member of a team will be rewarded. Two points will be deducted automatically if you are late for lab or miss the lab and have not contacted me with a darn good excuse (creativity is rewarded) PRIOR to the start of lab.

EXAMS: There will be three-hour exams and a final. The hour exams are 100 points each and the final is 200 points. The hour exams will be administered during the Thursday laboratory meeting of weeks 5, 10 and 14. The final exam is scheduled for Tuesday, May 17, 2005 from 14:45 – 16:45.

PROBLEM SETS: There will be several problem sets distributed during the semester. These problem sets will be handed out in class, along with the due dates. Each problem set is worth 20 points. There will be at least 5 problem sets during the semester.

TERM PAPER: Everyone will be assigned an instrumental technique on which to write a term paper. The paper must be at least 10 pages in length, and contain a minimum of 10 references, half of which must be from journal articles (not books) published in the last five years. The form of the paper and references will adhere to the requirements of the journal *Analytical Chemistry*. Term paper topics will be discussed during the first week of classes.

The term paper will be graded in three parts. Part I is an outline of the proposed paper. It will be due Friday, March 4 and should show the proposed organization of your paper by major and minor sub groupings. It is worth 10 points. Part II is a draft of your final paper. This draft will be due on Friday, April 1 and should contain the essentials of your final paper, including footnotes. A bibliography, properly formatted to the requirements of *Analytical Chemistry*, of articles used in preparing the draft must be included. The draft is worth 40 points, and the more complete the draft, the better the grade. One point will be deducted for every two typographical errors found in the draft. Errors in sentence structure or form will suffer a more extreme fate. I will comment on errors of omission or incompletely researched material and make suggestions for improvements for the final paper. Part III is the completed term paper. This paper must adhere to the manuscript requirements of *Analytical Chemistry* in terms of language used, organization, and formal style of footnotes and bibliography. In addition, 1 point will be deducted for **each** typographical error, and 10 points will automatically be deducted for an incomplete sentence. The final paper is worth 100 points. It is obviously to your advantage to read over your final paper carefully and to make good use of your word processor's spell checker. Errors of content, incorrect use of footnotes, grammar, etc. will affect the overall grade as will an assessment of readability. The final paper is due Friday, April 29. If time permits I will, at your request, read and comment on the content of the final paper prior to the final submission date. Further details about the term paper and how it will be graded will be provided at a later date.

GRADING: Grading will be based on total points, broken down as follows:

Assignment	Non – WE	WE
Hour Exams (3)	300	300
Problem Sets (5)	100	100
Laboratory Notebook/Reports	150	Lab Notebook WE
Laboratory Participation	50	50
Term Paper	150	WE
Final Exam	<u>200</u>	200
Total	950	WE Assignments <u>505</u>
		Total 1105

The final grade will be assigned based on a percentage of the possible points, with an A requiring 90%, a B requiring 80%, a C requiring 70% and a D requiring 60%. Actual grades may be "curved", but in no instance will any given letter grade require a higher score than listed above. **A grade of C or better in the course can only be achieved upon successful completion of all 10 laboratory experiments and submission of a report for each. If you earn less than 50% of the possible laboratory points, you will receive a course grade of F.** The + and - designations will be used for borderline scores.

ATTENDANCE: Attendance at classes is expected, and hopefully will be highly informative. You are responsible for all material discussed or assigned during class. **Laboratory attendance and participation is mandatory.** Missed labs may be made up only when a legitimate (or exceptionally creative) reason for the absence is presented. If you miss a lab and fail to make it up or complete all labs but do not submit your lab book for grading, you will receive a maximum grade of D. In addition, 2 points will be deducted from the laboratory participation grade for each day you are late for the pre-lab discussion at the beginning of the lab period unless the absence has been cleared with me in advance of class. Assigned experiments are to be conducted only during regularly scheduled class time unless prior arrangements are made with me.