

FCH 381 Instrumental Methods of Analysis

Dr. David J. Kieber

Syllabus **Spring, 2002**
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Week	Topic	Reading
1	Separations	Chapter 23
2	Chromatography	Chapter 23
3 (Lab)	High Performance Liquid Chromatography	Chapter 25
4 (Lab IC)	Other Chromatographic Methods	Chapter 26
5 (Lab)	Gas Chromatography	Chapter 24
6	Radiation and Matter	Chapter 19
6	Beer's Law	Chapter 19
7 (Lab)	UV/VIS and Fluorescence Spectroscopy	Chapters 19, 20, 21
8	Infrared Spectroscopy	Chapter 21
9, 10 (Lab)	Atomic Spectroscopy	Chapter 22
11 (Lab)	Inductively Coupled Plasma Spectroscopy	Reserve (see below)
12	Electrodes and Potentiometry	Chapter 15
13	Voltammetry	Chapter 18

Note: The problem set assigned for each chapter is given on page 3. The problems will not be handed in or graded. If you have difficulties with the problems see me during office hours.

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Text:

Harris, C.D. 1998. *Quantitative Chemical Analysis*. W.H. Freeman and Co., New York.

Moon Library Reserve Reading:

Boss, C.B. and K.J. Fredeen. 1999. *Concepts, Instrumentation and Techniques in Inductively Coupled Plasma Optical Emission Spectroscopy*. 2nd Ed. Perkin Elmer Corp., Norwalk, CT.

Exams:

Exam 1 (February 14), Exam 2 (March 26), Exam 3 (April 25), Final (TBA).

Three out of four exams will be used to determine the student's grade at the end of the semester. Each exam will be worth 33.3% of the exam grade (see below). The final exam will be cumulative including course and laboratory material for the entire semester.

Grading:

Lab Participation (5%), Lab Reports (50%), Exams (40%), Class Participation (5%)

Office Hours (417 or 441 Jahn):

Tuesday 1:00-4:00 pm and Thursday 11:00-12:00 am

Labs:

The lab will start in the second week of the semester. There will be six two-week labs. A lab report will be due the following Monday after each lab is finished. Refer to the attached handout on lab reports for information regarding the format of the reports. **The due dates for the labs are Monday: February 11 (Lab 1), February 25 (Lab 2), March 18 (Lab 3), April 1 (Lab 4), April 15 (Lab 5) and April 29 (Lab 6). Points will be deducted for lab reports that are handed in late.** There is no lab on January 17 or April 25.

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Problem sets from Harris, Quantitative Chemical Analysis, 5th Edition

Chapter	Problems
Handout	H₂O₂ regression analysis (see pg 4)
23	1, 7, 11, 15, 26, 28, 29, 32, 43
25	1a, 3, 6, 7, 9, 15
24	1, 2, 4, 5, 12, 17, 18, 19
4	20 (DL problem)
26	1, 2, 12, 14, 16, 17, 18, 21, 22, 24
19	1, 2, 3, 4, 6, 10, 11, 16, 18, 19, 23, 26
20	9, 17
21	13, 14, 17, 31, 32
22	1-7, 10, 11, 14, 16, 17, 19
15	5, 6, 13, 14, 21, 24, 26, 28, 34, 35, 37

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FCH 381 Homework Problem (H₂O₂ regression analysis)

- 1.) Given the following data for the calibration of hydrogen peroxide, use Excel (or other spreadsheet software) to plot a linear regression. In your analysis, determine the coefficient of determination (r^2), the slope (\pm SD) and the y-intercept (\pm SD). From the slope and y-intercept, determine the concentration of hydrogen peroxide in seawater, and the percent coefficient of variation (%CV).

H ₂ O ₂ addition (nM)	Fluorescence <u>Millivolt response</u>
seawater, no H ₂ O ₂ added	50.3
seawater, no H ₂ O ₂ added	50.9
seawater, no H ₂ O ₂ added	52.3
20.0	165.3
40.0	275.0
60.0	383.0
79.0	490.0
99.0	598.5
Reagent blank	9.2 millivolts

Note: %CV = (SD/mean)*100

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Lab Reports

Lab reports are to be handed in to me the Monday after each two-week lab is completed. Therefore, the lab reports are due on Mondays: **February 11 (Lab 1), February 25 (Lab 2), March 18 (Lab 3), April 1 (Lab 4), April 15 (Lab 5) and April 29 (Lab 6). Points will be deducted for lab reports that are handed in late. There is no lab on January 17 or April 25. Points will be deducted for lab reports that are late. There is no lab on April 25.** Your goal in writing the lab report is to clearly and concisely describe the experiment(s) that you conducted as well as the results that you obtained. A chemist should be able to use your lab report to successfully repeat your experiments. Therefore, when you write your reports, think about their content from the perspective of the reader. Each lab report should contain the following sections:

- 1.) Title Page** **On this page, you should identify the name of the lab, the class (FCH 381), your name and the name of your lab partners, and the date.**
- 2.) Introduction (25 pts)** This section should contain a brief background of the methodology used in the lab followed by a statement of the purpose and goals of the lab. Include pertinent chemical structures and reactions here.
- 3.) Method (25 pts)** In this section, you should reference the lab handout for details of the experimental method. In addition you should include the following sections **(a) standards** You should describe how solutions of the standards were prepared, if this information is not given (or is different) in the lab handout. **(b) apparatus** Provide a sketch of the instrument used, detailing the essential components. You should also list the instrumental settings that were generally used (e.g., scan speed, wavelength). **(c) procedure(s)** Describe any procedures that you employed that are different from that described in the lab handout (e.g., sample preparation/collection, derivatization reaction conditions, calibration methods). **(d)** Describe any sampling procedures (e.g., where the sample was collected and how it was collected).
- 4.) Results (25 pts)** The purpose of this section is to logically present your data and data analysis. Make sure to use text to present your results (do not only present numbers, calculations, etc). In all labs you should label your graphs (e.g., axes—with units, etc) and figures clearly and refer to them in the text. For example, show sample chromatograms here and label the peaks that you have identified, and identify the chromatograms with a figure number and a written description in the

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Lab Reports -- continued

results section. You should also show a graph(s) of your standard curve along with the results of your regression analysis. Include illustrative calculations to show how you derived values for calculated parameters (e.g., n). Also include sample calculations to show how concentrations of unknowns were determined. In your calculations, pay attention to the number of significant figures that you report. Also, be careful with your units. Finally, show the calculations for any error analysis that you performed. **Don't forget units and proper significant figures!!!** Present your results in a clear concise fashion (e.g., for the ion chromatography lab where you have five different standards, present the slope, and standard error data in one table for all five compounds).

5.) Discussion (25 pts) Here is where you interpret and discuss your results. What are potential sources of error in your lab (e.g., contamination)? When appropriate, how do your results compare to other investigators (i.e., literature values)-- are they significantly different? For example, in the spectroscopy lab, you are asked to compare your equilibrium constant with published values. You should also answer any questions posed at the end of your lab handout. If you detected unknowns that were not in your standard then discuss what they might be. How do your results from week 1 and week 2 compare (e.g., are the slopes of your standard curves significantly different?), etc.

Lab reports can be handwritten in your notebook (except for graphs) or typed as a separate lab report. If you submit hand written lab reports, I expect them to be legible. Points will be deducted if we cannot understand what you've written. Lab reports are expected to be handed in on time. Points will be deducted for late reports. Grading will be based on content (e.g., the quality of the data analysis and discussion sections) and clarity. For the lab reports, I expect you to know how to use Excel (available in the ESF computer cluster). Again, pay attention to significant figures. Also be sure to keep a good, detailed lab notebook. A well-written lab notebook simplifies that task of writing a lab report. It is also a permanent record of what you did in lab.