

**CHM 4130L
INSTRUMENTAL ANALYSIS LABORATORY
SPRING SEMESTER, 2012**

FACULTY MEMBER:

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**INSTRUMENTATION
SPECIALIST:**

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WEBSITE:

<http://lss.at.ufl.edu>

OBJECTIVES:

In CHM 4130L, students learn

- Fundamental concepts and practical usage of instrumentation for spectroscopic, chromatographic, electrochemical, thermal, and microscopic analysis.
- Calibration procedures for analysis of a variety of materials.
- Preparation of written reports and oral presentations.

REQUIRED MATERIALS:

- *Laboratory Manual for Instrumental Analysis* Spring 2012 (V. Young) and *Quantitation in Instrumental Analysis* © 2004 (K. R. Williams), both available at Target Copy. Additional reference materials and multimedia are on reserve at the Marston Science Library. You are also welcome to watch the DVDs in Leigh 202 or in your browser.
- Laboratory Notebook (with pre-numbered pages and carbon copies).
- Proper laboratory attire: long, loose-fitting pants, full shirt, shoes which cover the feet, departmentally-approved safety glasses, tie-back for long hair
- Miscellaneous photocopying throughout the semester
- one diskette, 3.5" or a flash drive

REQUIRED LABORATORY WORK: There will be a total of 11 laboratory assignments. Each group (two or three students per group) will complete one assignment per week, as specified on the experiment schedule. The 11 experiments are:

Basic Electronics
Electrochemical Methods
Microchemical Analysis
UV-Vis Spectrophotometry: Multicomponent Analysis
Liquid Chromatography
Ion Chromatography
Atomic Spectroscopy
Spectrometric Organic Analysis (MS, IR, NMR)
Molecular Fluorescence
Solid-Phase Microextraction and GC-MS
Surface Analysis (video-taped demonstration)

PRE-LAB QUIZ: Near the beginning of most laboratory periods, each student group will meet with one of the laboratory supervisors for an oral quiz on the experiment to be performed. The quiz will cover the lab

procedure (including the solutions to be prepared) and the instrument, as well as background material on the analytical technique. In preparation for the quiz, students should review the material in the experiment handout, including the experimental procedure and the suggested pre-lab questions. Reviewing the appropriate material from the CHM 4130 lecture or textbook, as well as CHM 4130L videos (available for viewing in Leigh 202, at the Marston Science Library Reserve Desk or through the course website) will be useful. Because the oral quiz will be “open lab book,” it is advisable to outline the planned experimental procedure and detail the solutions to be prepared in your lab notebook before coming to the lab. You may also refer to notes in your lab notebook regarding background material. The objective of the pre-lab quiz is to encourage each student to come to lab prepared to understand and perform the experiment efficiently. The Microchem pre-lab must be completed prior to the scheduled lab; this will be by arrangement. The electronics pre-lab is a written pre-lab due at the beginning of the class period.

HOMEWORK AND WRITTEN QUIZ: There will be one homework assignment due on 1/23,24/2012, and a written quiz on the Standard Additions Method at a mutually agreeable time on Friday, 2/3/2012.

ORAL REPORT: On April 2, 3 each student will give an oral report on a paper from an analytical chemistry journal. You must get your topic approved by March 19, 20, and you must turn in a paper copy of your paper to Dr. Young and your TA on March 26, 27.

LABORATORY REGULATIONS: Students must come to lab suitably dressed, as described under Required Materials. In the event of a laboratory accident requiring medical attention, the affected student(s) must go to the infirmary for treatment. Infirmary charges are the responsibility of the student.

After the experimental work is completed, glassware in the locker must be washed and the laboratory station left clean. If the group wishes to, the clean-up duties may be rotated among the group members. However, if the station is left dirty, points will be deducted from the subjective grade of each member of the group. If the experiment and clean-up are finished early, students are encouraged to begin their data reduction before leaving the lab, while the TA and lab computer are available.

LABORATORY REPORTS: Each student will be assigned to write reports or prepare worksheets for nine of the 11 experiments, as shown on the experiment schedule. For seven experiments (indicated as A/B in the schedule) all students will write reports. The other two experiments will be written up by one member of each team (A or B) or by two students in the groups of three. The number of points designated for each report is specified on the experiment schedule.

The report for each experiment (except for the electronics and the surface analysis experiments) is due at the start of the next laboratory period after the experiment is performed. The electronics report is the completion of a worksheet, which is turned in at the end of the experiment. The surface analysis is the identification of an unknown completed on the day of the experiment. The Microchemical Analysis report is written entirely in the laboratory notebook. For the written reports, each student must write his/her report in his/her own words. This means that all calculations, data analysis, and write-up must be done independently. Reports should be submitted on 8-1/2” x 11” paper and should include a cover sheet giving the title of the experiment, the date of the experiment and write-up, the name(s) of the lab partner(s) and the TA’s name. The required sections vary with experiment. The information below provides some general guidelines.

Purpose: This section should consist of one or two sentences giving a concise statement of the purpose of the experiment.

Procedure: If the procedure written in the notebook was used without alteration, this fact should be stated. Otherwise any deviations from the written procedure should be specified. **In addition, all instrument parameters, including the manufacturer and model number of the instrument, should be stated.**

Sample Calculations: This section should contain a sample of each type of calculation used in the data analysis. Units should be clearly expressed. Where the lab manual asks for calculations, they must be shown for each item, even if the method is the same. A concise header should be given for each sample calculation. For example: "Calculation of the diffusion current". Unless they are specifically requested, sample calculations of a statistical nature (standard deviation, linear regression, etc.) do not have to be shown.

Data and Results: This section should contain all data (weights, volumes, instrument readings, etc.) taken in the lab plus calculated results in tabular and, as required, graphical form. The tables and graphs must be prepared using a suitable spreadsheet program. One graph to a page, and it should fill the page. The entire Data & Results section should be preceded by one or more paragraphs explaining what is presented in the tables and graphs. Tables and graphs must each have a title and contain all pertinent data (e.g., concentrations of stock solutions, wavelengths, etc.) For the spreadsheet formulas – All numbers should have correct units and significant figures. Straight line graph data should be subjected to linear least squares analysis (included in the spreadsheet software). The original printer output should accompany the report of one member of the group. Refer to the Lab Manual for specific guidelines for data analysis for each experiment.

Conclusions: In this section the overall results of the analysis should be restated and, whenever possible, compared to literature values. Pertinent comments and observations about the results should be made and major sources of error (including sources in addition to personal errors) should be discussed. Include in this section the discussion requested in the lab manual for each experiment.

References: Experiment references should be cited using the format specified in JCE guideLitCited.pdf, available under Resources at our web site.

Proper citations for the lab manual are Young, V. *Laboratory Manual for Instrumental Analysis, Spring, 2012, Target Copy: Gainesville, 2012.*

Williams, K. R. *Quantitation in Instrumental Analysis, 2004.*

GRADING:

A. Division of Points

Homework (due 1/23,24/12)	75 points
Prelab Quizzes (10@ 35 points, none for Surface Analysis)	350 points
Reports (5 @ 125 pts, 3 @ 75 pts, 1 @ 50 pts)	900 points
Oral Report	75 points
Written Quiz (2/3/12)	50 points
<u>*Subjective</u>	<u>130 points</u>
Total Possible	1580 points

*The subjective grade will be based on attendance, preparation, teamwork, general attitude, lab area cleanup, and the completeness of the laboratory notebook.

B. Other Grading Policies

Late Reports: If a laboratory report is submitted late, 10% per day will be deducted. The maximum permissible late time is one calendar week. After that time, the report will be automatically graded as zero points. One "free late day" TOTAL will be granted to each student (not for Microchemical Analysis).

Unacceptable Reports: If a report is illegible or so poorly written that it cannot be graded, the instructor may require that the report be re-written. In this case, the report will be counted as "late" until it is acceptable.

Completion of Work: The absolute deadline for submission of reports is noon, Tuesday, 4/10/12.

STUDENTS WITH DISABILITIES: Appropriate accommodations will be provided, according to the policies at www.chem.ufl.edu/~itl/disabilities.html.

ACADEMIC HONESTY: Students are expected to obey the University of Florida Honor Code, detailed at www.chem.ufl.edu/~itl/honor.html. Violations will be reported to the Office of Student Judicial Affairs.

CHM 4130L EXPERIMENT SCHEDULE
REPORT AUTHORS AND POINT DESIGNATIONS
Spring 2012

GROUP→ DATE ↓	1	2	3	4
1/9,10	Electrochemical Methods (A/B, 125)			
1/17	Quantitation in Instrumental Analysis Homework			
1/23,24	Microchem (A/B, 75)	UV/Vis (A/B, 125)	LC (A, 125)	IC (B, 125)
1/30,31	UV/Vis (A/B, 125)	LC (A, 125)	IC (B, 125)	Microchem (A/B, 75)
2/6,7	LC (A, 125)	IC (B, 125)	Microchem (A/B, 75)	UV/Vis (A/B, 125)
2/13,14	IC (B, 125)	Microchem (A/B, 75)	UV/Vis (A/B, 125)	LC (A, 125)
2/20,21	Electronics (A/B, 75)	GC/MS (A/B, 75)	Fluor (B, 125)	ICP (A, 125)
2/27,28	GC/MS (A/B, 75)	Electronics (A/B, 75)	ICP (A, 125)	Fluor (B, 125)
3/5,6	SPRING BREAK			
3/12,13	ICP (A, 125)	Fluor (B, 125)	Electronics (A/B, 75)	GC/MS (A/B, 75)
3/19,20	Fluor (B, 125)	ICP (A, 125)	GC/MS (A/B, 75)	Electronics (A/B, 75)
3/26,27	Spec Organic (A/B, 125)			
4/2,3	Oral Reports (A/B, 75)			
4/9,10	TA evaluation and Surface Analysis (A/B, 50)			