

CHM 4130L, Instrumental Analysis Laboratory Summer 2016

Instructor:	Dr. Benjamin J. Killian, LEI 202A, 392-0528, killian@chem.ufl.edu Office Hours: W,R 2 nd pd.
Teaching Assistants:	TBA
Objectives:	CHM 4130L students learn: <ul style="list-style-type: none"> • Fundamental concepts and practical usage of instrumentation for spectroscopic, chromatographic, electrochemical, and mass-spectrometric analysis. • Calibration/quantitation procedures for analysis of a variety of materials. • Preparation of written and oral reports with basic statistical analysis.
Texts:	Killian, B. J. <i>Laboratory Manual for Instrumental Analysis</i> , Summer 2016, Target Copy: Gainesville, 2016. Williams, K. R. <i>Quantitation in Instrumental Analysis</i> , Target Copy: Gainesville, 2004.
Other Required Materials:	Laboratory Notebook with duplicate pre-numbered pages or composition book; safety glasses; departmentally approved attire (long, loose-fitting pants, full shirt, shoes which cover the feet, tieback for long hair); USB drive

Grade Distribution:

Written Quiz:	1 Standard Additions Quiz @ 50 pts (on 6/16)	50 pts.
Problem Set:	1 Standard Additions Problem Set @ 75 pts. (Due 5/26)	75 pts.
Written Reports:	1 @ 125 pts (Electrochem)	125 pts.
	2 @ 100 pts	200 pts.
	2 @ 75 pts	150 pts.
Project Proposal:	1 @ 100 pts	100 pts.
Project Report:	1 @ 150 pts	150 pts.
Pre-Lab Quizzes:	6 @ 35 pts (none for Electronics)	210 pts.
Data Grade:	5 @ 10 pts (none for Electronics, Microchem)	50 pts.
Subjective Grade:		100 pts.
Total		1210 pts.

Factors affecting the subjective grade will be the student's attendance record (lecture and lab), preparation for laboratory work, laboratory technique, cleanliness, understanding of the experiments, and general attitude. Ten (10) subjective points will be deducted for each unexcused absence. Random notebook "spot checks" will be conducted to check for properly written procedures and data collection.

Grading Scale :

<u>Letter Grade</u>	<u>Total Points</u>	<u>Letter Grade</u>	<u>Total Points</u>	<u>Letter Grade</u>	<u>Total Points</u>
A	≥1089	B-	≥932	D+	≥690
A-	≥1053	C+	≥871	D	≥653
B+	≥1016	C	≥811	D-	≥605
B	≥968	C-	≥774	E	<605

Pre-Lab Quizzes: Before the start of lab each week, all members of each group will meet together for a pre-lab quiz on the experiment to be performed. The quiz will cover the lab procedure (including solutions to be prepared) and the instrumentation, as well as background material on the analytical technique. In preparation for the quiz, students should review the material in the lab manual, including the experimental procedure and the suggested pre-lab questions. Reviewing the appropriate material from the CHM4130 lecture or textbook, as well as the CHM4130L videos will be useful. The videos are available at the Marston Science Library Reserve Desk or in Leigh 202 or on the department web page. Because the pre-lab quiz is "open notebook," you are encouraged to outline the planned experimental procedure and detail the solutions to be prepared in your lab notebook before coming to the pre-lab quiz. The electronics pre-lab will be a written pre-lab due at the beginning of the pre-lab. No pre-labs will be made-up; students who miss a pre-lab will receive a zero grade on the quiz.

The objective of the pre-lab quiz is to encourage each student to come to lab prepared to understand and perform the experiment efficiently.

Data Verification: One of the biggest stumbling blocks to 4130L students is timely preparation of lab reports. By putting off data analysis to the last minute students often present incomplete, incorrect, and poorly presented results and have little time to carefully construct report conclusions. Consequently, students are required to present their completed Sample Calculations and Data and Results for the Electrochemistry, UV/Vis, HPLC, ICP, and Molecular Fluorescence experiments to their TA for verification and approval by the end of day on the Friday immediately following the experiment, *even if the student is not responsible for writing the report*. This data verification will help to ensure correct results, and is worth 50 points total.

Written Reports: Each student is assigned to present reports for five of the seven experiments. All students are expected to write his/her report in his/her own words. All written reports (except Electronics and Microchem) are due at the start of the next lab period. The Electronics report consists of a worksheet to be completed in lab. The Microchem report is written in the notebook.

It is expected that reports will be neat and written in good English, with proper attention paid to paragraph structure, grammar, spelling, etc. Reports should be submitted on 8½" × 11" paper and should include a cover sheet giving the title of the experiment, the date of the experiment, the date of the write-up, the author's name (underlined), the names of all team-members, and the TAs' names. Except for equations, laboratory reports must be typed (double-space) using a minimum font size of 10 points. Please use a professional font, such as Times New Roman, Arial, Calibri, etc. The required sections for each report may vary with experiment; however, the information below provides some general guidelines.

I. Purpose: This section comprises a couple of sentences that give a concise statement of the purpose of the experiment and the methods used.

II. Experimental Procedure: If the procedure written in the notebook was followed without alteration, this fact should be stated and the lab manual cited. Otherwise, any deviations from the written procedure should be explained. In addition, ***all instrument/experimental parameters, including the manufacturer and model number, should be included.***

III. Calculations: Give a sample calculation with actual data (including units) for each type of calculation required in the data analysis. 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. Unless specified, you do not need to show calculations of a statistical nature (e.g., averages, standard deviations, linear regressions, etc.), but you do need to show percent error and percent difference calculations when comparing results.

IV. Data and Results: This section should contain all data (weights, volumes, instrument readings, etc.) taken in the lab, plus calculated results in tabular and, if required, graphical form. Use a spreadsheet program (e.g., Excel) to prepare tables and graphs. *Format graphs such that they fill a single page.* The entire Data and Results section should be preceded by one or more paragraphs explaining what is presented in the tables and figures. Tables and graphs must each have a title and contain all pertinent data (e.g., concentrations of stock solutions, wavelengths, etc.). All values should have units and be presented to the correct number of significant figures. Straight line data should be subjected to least squares analysis (included in the spreadsheet software). The original output from the experiment must be included in the report of one member of the team.

VII. 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 error) should be discussed. Include in this section any discussion requested in the lab manual for each experiment. Each report should include a detailed discussion of both random and systematic errors that could have occurred with the instrumental method being used (at least three sources of each). All systematic errors must include a discussion of how the error would affect the final results.

VIII. References: Special procedures, literature values, and discussions of previous research results must be referenced in the text using superscript numbers. The references themselves belong in a separate section at the end of the report using the format specified in the *ACS Style Guide* (see <https://www.libraries.psu.edu/content/dam/psu/psu/psu/pams/documents/QuickGuideACS.pdf> for details). Note: The laboratory manual should be referenced as shown on the first page of this syllabus.

Additional Grading Policies: Written reports must be submitted by 12:30 PM on the designated dates. All assignments submitted after 12:30 PM are considered late. Deductions at the rate of 5% per day (including weekends) will be assessed for late work. The maximum allowable late time is one calendar week, after which a grade of zero will be assigned. All written work (late or otherwise) must be received by 12:30 PM on Thursday, 4 August 2016.

Schedule of Experiments and Reports: Each team member will be assigned a letter code (either A or B). For those experiments denoted by the A or B, the report is to be submitted by that team member. If there is no letter designation, then all team members will submit reports.

Laboratory Schedule			
Date	Experiment		Date Due
	Team 1	Team 2	
5/12	Electrochemistry 125 pts		5/19
5/19	Electronics* 75 pts	Microchem [†] 75 pts	
5/26	Microchem [†] 75 pts	Electronics* 75 pts	
6/2	UV/Vis (A) 100 pts	HPLC (B) 100 pts	6/9
6/9	HPLC (B) 100 pts	UV/Vis (A) 100 pts	6/16
6/16	Project Proposal [‡] 100 pts		6/30
6/23	No Lab Summer Break		
6/30	ICP (B) 100 pts	Fluorescence (A) 100 pts	7/7
7/7	Fluorescence (A) 100 pts	ICP (B) 100 pts	7/14
7/14	Project Prep and Construction		
7/21	Project Construction		
7/28	Project Completion		
8/4	Project Report		

*The electronics worksheet will be submitted during lab time.

†The Microchemistry report is written in the notebook. The notebook will be submitted the day following the experiment.

‡Each team will submit a single proposal.

Class Attendance: Requirements for class attendance and make-up assignments are consistent with university policies that can be found at <https://catalog.ufl.edu/ugrad/current/regulations/info/attendance.aspx>.

Students with Disabilities: Students with disabilities requesting accommodations should first register with the Disability Resource Center (352.392.8565, www.dso.ufl.edu/drc/) by providing appropriate documentation. Once registered, students will receive an accommodation letter, which must be presented to the instructor when requesting accommodations. Students with disabilities should follow this procedure as early as possible in the semester.

Grading Policies: Information about the most current UF grading policies for the assigning of grade points can be found at <https://catalog.ufl.edu/ugrad/current/regulations/info/grades.aspx>.

Course Evaluations: Students are expected to provide feedback on the quality of instruction in this course by completing on-line evaluations at <https://evaluations.ufl.edu>. Evaluations are typically open during the last two or three weeks of the semester, but students will be given specific times when they are open. Summary results of these assessments are available to students at <https://evaluations.ufl.edu/results/>.

Academic Honesty: UF students are bound by The Honor Pledge, which states, "We, the members of the University of Florida community, pledge to hold ourselves and our peers to the highest standards of honor and integrity by abiding by the Honor Code." On all work submitted for credit by students at the University of Florida, the following pledge is either required or implied: "On my honor, I have neither given nor received unauthorized aid in doing this assignment."

Sharing of lab reports between students is considered a violation of the Honor Code, as is the sale or transfer of graded or ungraded course materials to another student for use in this course (current or future semesters). All violations will be reported to the Office of Student Judicial Affairs.

Additional information may be found at <https://www.dso.ufl.edu/sccr/process/student-conduct-honor-code/>. If you have specific questions, please consult with the instructor or your TA.

Student Wellness: We hold student success very highly, and we hope that any concerns of both an academic and a personal nature can be addressed in a timely and helpful manner. The Counseling and Wellness Center is an important service provided by the university to help with a variety of personal issues and concerns. The center can be contacted at 352.392.1575. More information is available at <http://www.counseling.ufl.edu/cwc/Default.aspx>.