

## CHM 4130L, Instrumental Analysis Laboratory

### Fall 2016

**Instructor:** Dr. Kathryn R. Williams, CLB 220, 392-7369, [krw@chem.ufl.edu](mailto:krw@chem.ufl.edu)  
Office Hours: W, 8<sup>th</sup>; F, 6<sup>th</sup>

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**Objectives:** 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 with basic statistical analysis.

**Texts:** Williams, K.R.. *Laboratory Manual for Instrumental Analysis*, Fall, 2016, Target Copy: Gainesville, 2016.

**Other Required Materials:** Laboratory Notebook with duplicate pre-numbered pages; safety glasses; departmentally approved attire (long, loose-fitting pants, full shirt, shoes which cover the feet, tieback for long hair); diskette or memory stick

#### Grade Distribution:

Written Quiz:	1 Standard Additions Quiz @ 50 pts (T, 9/6)	50 pts.
Problem Set:	1 Standard Additions Problem Set @ 75 pts. (Due W, 8/31, at noon)	75 pts.
Written Reports:	4 @ 100 pts (UV/Vis, ICP or MF, Spec Org, HPLC)	400 pts.
	5 @ 75 pts (Microchem, IC or FTIR, Electronics, Electrochem I and II)	375 pts.
Pre-Lab Quizzes:	10 @ 35 pts (none for Electronics)	350 pts.
Lab Practical:	1 @ 35 pts on Electronics (10/17,18)	35 pts.
Subjective Grade:		100 pts.
<b>Total</b>		<b>1385 pts.</b>

Factors affecting the subjective grade will be the student's attendance record, 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** (in % using usual rounding conventions for fractions):

<u>Letter Grade</u>	<u>Percentage</u>	<u>Letter Grade</u>	<u>Percentage</u>	<u>Letter Grade</u>	<u>Percentage</u>
A	>90	B-	>77	D+	>57
A-	>87	C+	>72	D	>54
B+	>84	C	>67	E	<54
B	>80	C-	>64		

Note: Chemistry majors earning grades below C (C-, D+, D, or E) must repeat the course to earn credit towards the degree.

**Pre-Lab Quizzes:** Before or during lab each week, all members of each group will meet together for a pre-lab quiz on the experiment to be performed. 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 quiz will cover the lab procedure (including 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 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 <https://www.chem.ufl.edu/undergraduate/courses-and-curriculum/chemistry-laboratories/analytical/>. 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. No pre-labs will be made-up; students who miss a pre-lab will receive a zero grade on the quiz.

**Written Reports:** Each student is assigned to prepare reports for nine of the ten experiments. You are expected to write your report in your own words (i.e., all calculations, data analysis, and write-up must be done independently). The experiment assignments are provided on the experiment schedule. Those experiments marked A/B will be written by all students. Those marked either A or B will be written by a subset of the students in each team (assigned either A or B). The points designated for each report are denoted on the experiment schedule.

All written reports (except Microchemistry, Electronics, and Spec Organic Anal) are due at the start of the next lab period. The Electronics report consists of a worksheet to be completed and submitted at the end of the laboratory period. The Microchemistry report is written entirely in the laboratory notebook and submitted at the end of the laboratory period. For Spec Organic Analysis, the full report is due by noon, Wed, October 19.

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’ name. Except for equations, laboratory reports must be typed (double-space) using a minimum 12-point font size. Please use a professional font, such as Times New Roman or Arial. 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 (e.g., ICP and MF reports and Standard Additions problem set), 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 with 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* (<http://pubs.acs.org/isbn/9780841239999>). Note: The laboratory manual should be referenced as shown on the first page of this syllabus.

**Electronics Practical:** During the lab periods on 10/17,18, students will meet individually with Dr. Williams for a 15-20 minute practical exam on the electronics experiment. The material will be directly related to the circuits and other concepts in the experiment. You will be expected to wire at least one of the circuits by yourself and answer questions about it.

**Additional Grading Policies:** Written reports must be submitted by 12:50 PM on the designated dates. All assignments submitted after 12:50 PM will be 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 12:50 PM on Wednesday, 12/7/16.

Any reports that are deemed substandard or ungradable will be returned to the student for revision and resubmission. The standard late deductions will apply.

#### **Students with Disabilities:**

Students with disabilities requesting accommodations should first register with the Disability Resource Center (352-392-8565, [www.dso.ufl.edu/drc/](http://www.dso.ufl.edu/drc/)) by providing appropriate documentation.

#### **Academic Honesty:**

The Honor Code (<http://www.dso.ufl.edu/sccr/process/student-conduct-honor-code/>) specifies a number of behaviors that are in violation of this code and the possible sanctions. Furthermore, you are obligated to report any condition that facilitates academic misconduct to appropriate personnel. If you have any questions or concerns, please consult with the instructor or TAs in this class.

The sale or transfer of graded or ungraded course materials to another student for use in this course (current or future semesters) is in violation of the Honor Code. All violations will be reported.

#### **Online Evaluations:**

Students are expected to provide feedback on the quality of instruction in this course by completing online 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/>.

Date	Experiment				Date Due
	Team 1	Team 2	Team 3	Team 4	
8/22,23	Electrochemistry I (A/B) 75 pts				8/29,30
8/29,30 <sup>1</sup>	Electrochemistry II (A/B) 75 pts				9/6
9/5, 6	Standard Additions Quiz 50 pts				N/A
9/12,13	HPLC (A/B) 100 pts	UV-Vis (A/B) 100 pts	Microchem <sup>2</sup> (A/B) 75 pts	Electronics <sup>3</sup> (A/B) 75 pts	9/19,20
9/19,20	Electronics <sup>3</sup> (A/B) 75 pts	HPLC (A/B) 100 pts	UV-Vis (A/B) 100 pts	Microchem <sup>2</sup> (A/B) 75 pts	9/26,27
9/26,27	Microchem <sup>2</sup> (A/B) 75 pts	Electronics <sup>3</sup> (A/B) 75 pts	HPLC (A/B) 100 pts	UV/Vis (A/B) 100 pts	10/3,4
10/3,4	UV/Vis (A/B) 100 pts	Microchem <sup>2</sup> (A/B) 75 pts	Electronics <sup>3</sup> (A/B) 75 pts	HPLC (A/B) 100 pts	10/10,11
10/10,11	Spectrophotometric Organic Analysis (A/B) 100 pts				Noon, Wed, 10/19
10/17,18	Electronics Practical 35 pts				N/A
10/24,25	FTIR (A) 75 pts	MF (B) 100 pts	ICP (A) 100 pts	Ion Chrom (B) 75 pts	10/31;11/1
10/31;11/1	Ion Chrom (B) 75 pts	ICP (A) 100 pts	MF (B) 100 pts	FTIR (A) 75 pts	11/7,8
11/7,8	ICP (A) 100 pts	Ion Chrom (B) 75 pts	FTIR (A) 75 pts	MF (B) 100 pts	11/14,15
11/14,15	MF (B) 100 pts	FTIR (A) 75 pts	Ion Chrom (B) 75 pts	ICP (A) 100 pts	11/21,22
11/21,22	No Lab				N/A
11/28,29	Tour of Mass Spec Facilities				N/A
12/5,6	No Lab				N/A

<sup>1</sup> The Standard Additions Problem Set must be submitted to Dr. Williams by Wed, 8/31, at noon.

<sup>2</sup> The report for the Microchemistry experiment will be written in the notebook and submitted during the lab period.

<sup>3</sup> The Electronics worksheet will be submitted during the lab period.