

## CHM 4130L, Instrumental Analysis Laboratory Fall 2024

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**Objectives:** CHM 4130L students learn:

- Fundamental concepts and practical usage of instrumentation for spectroscopic, chromatographic and mass spectrometric analysis.
- Calibration procedures for analysis of a variety of materials.
- Preparation of written reports.

**Other Required Materials:** Laboratory Notebook; safety glasses;  
departmentally approved attire (long, loose-fitting pants, full shirt, shoes which cover the feet, tieback for long hair); flash drive

### Grade Distribution:

Written Reports:	9 @ 100 pts	900 pts.
Pre-Labs:	9 @ 45 pts	405 pts.
Final Lab Practical		240 pts
<b>Total</b>		<b>1545 pts.</b>

**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	≥93	B-	≥80	D+	≥67
A-	≥90	C+	≥77	D	≥63
B+	≥87	C	≥73	D-	≥60
B	≥83	C-	≥70	E	<60

Note: Chemistry majors earning grades below C (C-, D+, D, or E) must repeat the course to earn credit towards the degree. A curve may be applied at the end of the semester.

**Pre-Labs:** Each week, students will be given an oral prelab exam before the start of the experiment. This exam is designed to ensure the students have read the lab material beforehand and have a basic understanding of how the instrument operates. Guideline questions will be given out in the lab write ups for students to use to study. Many of these questions will require the students to look up reference material (most of which will be cited in the prelab) in order to answer the questions. These are designed so that students are prepared for the lab ahead of time and give them an idea of the strengths and limitations of the experiments and instrumentation. No make-up prelabs will be allowed

**Written Reports:** Each student will write reports for the 9 experiments and the final practical. 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. All lab reports will be full reports. However, for the first 4 labs, grading emphasis will be on which section is in parentheses below the experiment. For example, Team 1 is doing ICP-AE the first week, so the introduction will be weighed more in that report.

All written reports are due at the start of the next lab period on Canvas.

It is expected that reports will be neat and written in good English, with proper attention paid to paragraph structure, grammar, spelling, etc. Reports will be submitted on Canvas and should include the title of the experiment, the date of the experiment, the date of the write-up, the author's name (underlined), and the TAs' name. Laboratory reports must be typed (double-spaced) using a minimum 12-point font size in a font such as Times New Roman, Calibri or Arial. The information below provides some general guidelines.

**Abstract:** A very short (250 words or less) summary of the lab report. This covers the purpose, results and discussion and conclusion. No citations should take place here. For the shorter lab reports, just state the purpose, briefly.

**Introduction:** Give a brief background on the instrument and instrumental method that you used in the lab. Additionally, you should also discuss the sample that you are measuring. Finally, you will sum this up as the purpose of the lab. Regardless of the lab report, anything that is not common knowledge must be cited. For the longer lab reports, you will need to go into more detail.

**Experimental Procedure:** The entire lab procedure should be reproduced in your own words. All parameters should be noted (e.g., concentrations of stock solutions, wavelengths, etc) and any model numbers should be included. The goal is to write this in a way so that someone can read your lab report and reproduce your experiment without issue. **DO NOT INCLUDE:** Turn the instrument on, click on the start icon, and the like

Formulas used in calculations should be shown here. You do NOT have to show an example. You are adults, I can safely assume you can use calculators and Excel. However, it would not hurt to have your calculations written in your lab notebook in case you get stuck.

**Results and Discussion:** This is two sections combined into one. You will start by stating your results and referencing your graphs and tables of the data. You comment on any outlying data and any points removed. If there was a calibration curve, be sure to comment on the linearity of the curve. Any statistical data (such as standard deviation) should also be mentioned. You will also discuss what your results mean. If your signal changed when you added another component, why did that happen? Explain the peaks in spectra or chromatograms. You will need to explain if your results are correct or what your unknowns could possibly be. Any error analysis should be included in this section. It is likely you will have to make a couple of literature citations in this section. Any questions asked in the lab write up should be answered here.

Tables and graphs should appear in this section. Tables should include any values obtained through measurement, calculation or data acquisition. Tables should be preceded by "Table 1: Some descriptor of the data". Each column should have a title (with units if appropriate) and be center justified. The left most column, with the identifying name, should be left justified. All numerical data should be center justified and have appropriate sig figs. Graphs, either printout spectra or made through excel should have proper axes labels, units and the correct number of significant figures. Trendlines (linear regressions) and the coefficient of determination ( $R^2$ ) should be shown. If multiple measurements were taken, then standard deviation should be shown where appropriate. Graphs should be followed by "Figure X: Short explanation of the figure"

**Conclusions:** Restate the purpose of the experiments and the results obtained. Any errors in your results compared to literature values/answers should be repeated as well. This should be kept rather short (paragraph or two at the most)

**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.

**Lab Practical Final:** The final lab in this course will have you determining an unknown using the various instrumental techniques throughout the semester. Any instrumentation used will be valid for determining your unknown.

**Lab Manual:** All lab instructions will be posted to Canvas. Please read them before class. You may use a tablet, laptop or printout to view the experimental procedure.

**Attendance:** Attendance is required at all scheduled laboratory periods, unless you are informed otherwise by your TA or the instructor. Make ups will be granted only when justified. If you know ahead that you will have to miss lab, notify your TA and Dr. Jacobs in advance. If you are sick and cannot reach anyone before lab, you will have to present written evidence of the illness. **If you are not feeling well, do not come to lab.**

**Additional Grading Policies:** Written reports must be submitted by 12:50 PM on the designated dates. All assignments submitted after will be considered late. Deductions at the rate of 5% per day (including weekends) off the final score 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 3:00 PM on December, 12/6/23.

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 instructors 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 professional and respectful feedback on the quality of instruction in this course by completing course evaluations online via GatorEvals. Guidance on how to give feedback in a professional and respectful manner is available at <https://gatorevals.aa.ufl.edu/students/>. Students will be notified when the evaluation period opens, and can complete evaluations through the email they receive from GatorEvals, in their Canvas course menu under GatorEvals, or via <https://ufl.bluera.com/ufl/>. Summaries of course evaluation results are available to students at <https://gatorevals.aa.ufl.edu/public-results/>.

**Course Fees:**

You've already paid this, but we are now required to say that your fee for this course is \$85.00.

Date	Experiment				Date Due
	Team 1	Team 2	Team 3	Team 4	
8/26,27	Lab lecture on writing lab reports				N/A
9/2,4	No lab (Labor Day)				
9/9,10	ICP-AE (Intro)	FTIR (Experimental)	UPLC-MS (Results and Discussion)	UV/Vis (Abstract and Conclusion)	9/16,17
9/16,17	FTIR (Experimental)	UPLC-MS (Results and Discussion)	UV/Vis (Abstract and Conclusion)	ICP-AE (Intro)	9/23,24
9/23,24	UPLC-MS (Results and Discussion)	UV/Vis (Abstract and Conclusion)	ICP-AE (Intro)	FTIR (Experimental)	9/30,10/1
9/30,10/1	UV/Vis (Abstract and Conclusion)	ICP-AE (Intro)	FTIR (Experimental)	UPLC-MS (Results and Discussion)	10/7,8
10/7,8	Fluorescence	IC	NMR	MALDI	10/14,15
10/14,15	IC	NMR	MALDI	Fluorescence	10/21,22
10/21,22	NMR	MALDI	Fluorescence	IC	10/28,29
10/28,29	MALDI	Fluorescence	IC	NMR	11/4,5
11/4,5	Class GC lab				11/18,19
11/11,12	No lab, Veteran's Day				
11/18,19	Lab Practical				
11/25,26	No Lab, Thanksgiving Break				
12/2,3	Turn in practical (Back up week)				