

CHM 4130L, Instrumental Analysis Laboratory Spring 2018

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Objectives: CHM 4130L students learn:

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

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); flash drive

Grade Distribution:

| | | |
|----------------------------|--|------------------|
| Written Reports: | 5 @ 100 pts (UV/Vis, ICP-AE, FTIR, HPLC, Mass Spec) | 500 pts. |
| | 5 @ 75 pts (IC, Fluorescence, Electronics I and II, Electrochem) | 375 pts. |
| Pre-Labs: | 10 @ 35 pts (none for Electronics I) | 350 pts. |
| Electronics Practical: | 1 @ 35 pts | 35 pts. |
| Notebook/Subjective Grade: | | 100 pts. |
| Final Lab Practical | | 140 pts |
| Total | | 1500 pts. |

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 | ≥93 | B- | ≥80 | D+ | ≥57 |
| A- | ≥90 | C+ | ≥77 | D | ≥53 |
| B+ | ≥87 | C | ≥62 | E | <53 |
| B | ≥82 | C- | ≥60 | | |

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

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 writes reports for 9 of the 11 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. Unlike previous courses, some of your lab reports will be in the style of a manuscript. You will be submitting these full lab reports for the 100 pt lab (UV/Vis, ICP, FTIR, Mass Spec, HPLC and the Final Project). You will submit shorted lab reports for the 75 pt labs (IC, MF, Electrochem, Electronics I and II). Details on what is required for each report is below.

All written reports (except Electronics) are due at the start of the next lab period. The Electronics reports consists of a worksheet to be completed and submitted at the end of the laboratory periods.

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 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-spaced) using a minimum 12-point font size in a font such as Times New Roman, Calibri or Arial. The required sections for each report may vary with experiment; however, 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.

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.

Experimental Procedure: For the short lab reports, 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.

For the long lab reports, 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.

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: For the short reports, simply state your results in paragraph form and how you arrived at these results. Provide a short explanation for error.

For the long reports: 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.

Electronics Practical: During the lab periods on 3/12,13 students will meet individually with the instructor 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.

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. More information about the report for this will be given as the practical date approaches.

Lab Manual: All lab instructions will be posted to Canvas. Either download the manual and print it off/load it on a tablet or write out the procedure in your lab notebook. You must have the lab instructions BEFORE entering lab.

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) 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 12:50 PM on Wednesday, 4/25/18.

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/) 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 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/>. The instructor highly values this feedback so that he can adjust the course for later semesters.

| Date | Experiment | | | | Date Due |
|---------|------------------------------------|----------------|----------------|----------------|----------|
| | Team 1 | Team 2 | Team 3 | Team 4 | |
| 1/8,9 | Lecture on Lab Reports | | | | N/A |
| 1/15,16 | No Lab | | | | |
| 1/22,23 | Electrochemistry and Electronics I | | | | 1/29,30 |
| 1/29,30 | Electrochemistry and Electronics I | | | | 2/5,6 |
| 2/5,6 | HPLC | UV/Vis | Electronics II | FTIR | 2/12,13 |
| 2/12,13 | FTIR | HPLC | UV/Vis | Electronics II | 2/19,20 |
| 2/19,20 | Electronics II | FTIR | HPLC | UV/Vis | 2/26,27 |
| 2/26,27 | UV/Vis | Electronics II | FTIR | HPLC | 3/12,13 |
| 3/3,4 | No Lab Have a safe Spring Break | | | | |
| 3/12,13 | Electronics Practical | | | | |
| 3/19,20 | ICP-AE | UPLC/MS | Fluorescence | IC | 3/26,27 |
| 3/26,27 | IC | ICP-AE | UPLC/MS | Fluorescence | 4/2,3 |
| 4/2,3 | Fluorescence | IC | ICP-AE | UPLC/MS | 4/9,10 |
| 4/9,10 | UPLC/MS | Fluorescence | IC | ICP-AE | 4/16,17 |
| 4/16,17 | Lab Practical | | | | 4/22,23 |
| 4/23,24 | Tour of Mass Spec Facility | | | | |

The Electronics worksheets will be submitted during the lab period.