

CHM4130L, Instrumental Analysis Laboratory Spring 2014

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| Instructor: | Dr. Benjamin J. Killian, LEI 202A, 392-0528, killian@chem.ufl.edu Office Hours: M 4th Per.; W, 6th Per.; R, 3rd Per.; F 4th Per. |
| Teaching Assistants: | Gelin Looi, LEI 310, gelin@chem.ufl.edu Liz Dhummakupt, CLB 200, muffy@chem.ufl.edu |
| Instrumental Specialist: | Dr. Oleg Matveev, LEI 200B, 846-0840, oleg@chem.ufl.edu |
| Objectives: | CHM 4130L students learn: <ul style="list-style-type: none"> • 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 and oral reports with basic statistical analysis. |
| Texts: | Killian, B. J. <i>Laboratory Manual for Instrumental Analysis</i> , Spring 2014, Target Copy,; Gainesville, 2014 Williams, K. R. <i>Quantitation in Instrumental Analysis</i> , Target Copy: Gainesville, 2004 |
| 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); USB drive. |

Grade Distribution:

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|-------------------|--|-----------------|
| Problem Set: | 1 Standard Additions Problem Set @ 75 pts (Due 1/21) | 75 pts |
| Written Quiz: | 1 Standard Additions Quiz @ 50 pts | 50 pts |
| Written Reports: | 1 @ 125 pts (Electrochem) | 125 pts |
| | 3 @ 100 pts (UV/Vis or HPLC; ICP or MF; Spec Org) | 300 pts |
| | 2 @ 75 pts (Microchem and Electronics) | 150 pts |
| | 1 @ 50 pts (Surface Analysis) | 50 pts |
| Oral Reports: | 2 @ 75 pts (IC and GC/MS) | 150 pts |
| Pre-Lab Quizzes: | 10 @ 35 pts (none for Surface Analysis) | 350 pts |
| Data Grade: | 7 @ 10 pts (Electrochem; UV/Vis; HPLC; IC; GC/MS; ICP, MF) | 70 pts |
| Subjective Grade: | | 100 pts |
| Total | | 1440 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:

| Letter Grade | Percentage | Letter Grade | Percentage | Letter Grade | Percentage |
|--------------|------------|--------------|------------|--------------|------------|
| 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 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 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 the Marston Science Library Reserve Desk or in Leigh 202. 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 is 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, GC/MS, HPIC, 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 70 points total.

Written Reports: Each student is assigned to present reports for nine of the eleven experiments. All students are expected to write his/her report in his/her own words (i.e., all calculations, data analysis, and write-up must be done independently). These assignments are provided on the experiment schedule. Those experiments marked A/B (with the exception of the IC and GC/MS experiments) 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 Surface Analysis) are due at the start of the next lab period. The Electronics report consists of a worksheet to be completed in lab. The Surface Analysis experiment involves the identification of an unknown completed on the day of the experiment. The Microchemistry report is written entirely in the laboratory 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 8 1/2 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 authors 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 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, 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. Guidelines for tables and graphs are given in Young, V. *Laboratory Manual for Introductory Analytical Chemistry*, pp 5-8 (and older versions, as well). 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.
- V. **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.
- VI. **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, which can be found at <http://jchemed.chem.wisc.edu/Journal/Authors/References.html>. Note: The laboratory manual should be referenced as shown on the first page of this syllabus.

Oral Reports: You will present two oral reports during the semester (IC and GC/MS). You will need to prepare your report as “slides”, printed in landscape, to be fastened to a presentation board (à la science fair). You will include pertinent parts of each of the sections listed above and present the information to Dr. Killian. You will be given 20 minutes to present the report, 15 - 17 minutes for presentation and 3 - 5 minutes for questions.

Additional Grading Policies: Written reports must be submitted by 12:50 PM on the designated dates. All assignments submitted after 12:50 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 12:50 PM on Wednesday, 04/23/2014.

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

Students with Disabilities: Appropriate accommodations will be provided, according to the policy 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.

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.

Schedule of Experiments and Reports:

| Laboratory Schedule | | | | | |
|---------------------|--|--|--|--|----------|
| Date | Experiment | | | | Date Due |
| | Team 1 | Team 2 | Team 3 | Team 4 | |
| 1/6,7 | Electrochemistry (A/B) 125 pts | | | | 1/13,14 |
| 1/13,14 | FTIR (A/B) 100 pts | | | | 1/24 |
| 1/20,21 | NO LAB (MLK Day) | | | | |
| 1/27,28 | Electronics (A/B) 75 pts | Microchem [†] (A/B) 75 pts | UV/Vis (A) 100 pts | HPLC (B) 100 pts | 2/3,4 |
| 2/3,4 | Microchem [†] (A/B) 75 pts | UV/Vis (A) 100 pts | HPLC (B) 100 pts | Electronics (A/B) 75 pts | 2/10,11 |
| 2/10,11 | UV/Vis (A) 100 pts | HPLC (B) 100 pts | Electronics (A/B) 75 pts | Microchem [†] (A/B) 75 pts | 2/17,18 |
| 2/17,18 | HPLC (B) 100 pts | Electronics (A/B) 75 pts | Microchem [†] (A/B) 75 pts | UV/Vis (A) 100 pts | 2/24,25 |
| 2/24,25 | Spec Organic (A/B) 100 pts | | | | 3/10,11 |
| 3/3,4 | NO LAB (Spring Break) | | | | |
| 3/10,11 | Standard Additions Quiz | | | | |
| 3/17,18 | IC [‡] (A/B) 75 pts | MF (A) 100 pts | ICP (B) 100 pts | GC/MS [‡] (A/B) 75 pts | 3/24,25 |
| 3/24,25 | MF (A) 100 pts | ICP (B) 100 pts | GC/MS [‡] (A/B) 75 pts | IC [‡] (A/B) 75 pts | 3/31,4/1 |
| 3/31,4/1 | ICP (B) 100 pts | GC/MS [‡] (A/B) 75 pts | IC [‡] (A/B) 75 pts | MF (A) 100 pts | 4/7,8 |
| 4/7,8 | GC/MS [‡] (A/B) 75 pts | IC [‡] (A/B) 75 pts | MF (A) 100 pts | ICP (B) 100 pts | 4/14,15 |
| 4/14,15 | Surface Analysis* | | | | |
| 4/21,22 | NO LAB | | | | |

[†] The report for the Microchemistry experiment will be written in the notebook and submitted during the lab period.

[‡] Oral reports must be scheduled with Dr. K before leaving lab.

* The report for the Surface Analysis experiment will be submitted during the lab period.