Colorado State University
Spring Session 2024

CHEM 431
Instrumental Analysis Laboratory
Supplementary Syllabus

Laboratory Location: Yates 503

Instructor: Dr. Joseph DiVerdi
Email Address: joseph.diverdi@colostate.edu
Office Location: Yates 504A
Office Phone: 970.980.5868
Office Hours: M 3:00 PM – 4:00 PM
R 1:00 AM – 12:00 PM
M 3:00 PM – 4:00 PM or by appointment
Office Hour Location: Yates 501, 502, 503, 504

Graduate Teaching Assistant: Ricky Peña
Email Address: ricardo.pena@colostate.edu
Office Hours: WF: 2:00 PM – 3:00 PM
Office Hour Location: Yates 503

Section #: L01 **Laboratory Hours**: W: 12:00 AM – 3:00 PM
Section #: L02 **Laboratory Hours**: W: 3:00 PM – 6:00 PM

Course Objectives: The objectives of this laboratory component of the course are: (1) To provide the student with practical experience in the techniques of experimental, instrument-oriented, analytic chemistry. (2) To deepen the student's understanding of the underlying principles of the measurement by various laboratory instruments. (3) To develop more sophisticated instrument skills in the student. (4) To increase the student's ability to communicate effectively and professionally both in writing and in speech.

Course Topics: The experimental measurement including quantitation of chemical properties using spectroscopy, chromatography and electrochemistry. The application of experimental physical chemistry methods. The development of instrumental methods. The creation, visualization, processing, analysis and critical assessment of quantitative analytic data. Making a $%#& instrument work properly.

Prerequisites: CHEM 232 - Quantitative Analysis Laboratory - previous enrollment and CHEM 372 - Physical Chemistry I - either previous or concurrent enrollment.

Textbook: No textbook is required. Textbooks which should be on the book shelves or in some disk directory of aspiring chemists: *Principles of Instrumental Analysis*, Douglas Skoog, F. James Holler and Stanley Crouch, Sixth Edition, McGraw-Hill, ISBN 9780495012016; *An Introduction to Error Analysis*, John Taylor, University Science Books, ISBN 9780935702750.

Academic Policies & Integrity: The University's policies on Students' Responsibilities and Rights including Personal Integrity, Academic Integrity and related areas are presented at http://catalog.colostate.edu/, especially as described in Section 1.6 on "Policies and Guiding Principles". These policies will be followed in this course. The short of it is compactly contained in the Honor Pledge:

"*I will not give, receive or use any unauthorized assistance.*"

Assistance from colleagues, experts and other sources is authorized but a threshold exists above which the assistance must be cited. This will be discussed (at some length) in the laboratory sessions.

Additional information on University policies on course registration, add/drop, withdraw and incomplete grades can be found at http://www.catalog.colostate.edu/, especially as described in Section 1.7 on "Advising and Registration". These policies will be followed in this course also.

Web-based Course Tools: http/:canvas.colostate.edu/. As a registrant of this course, the student can access its site and must do so to view announcements, receive grades and submit laboratory report and presentation files. Experimental descriptions are available at http://www.chem.colostate.edu/diverdi/.

Course Plan & Organization: This laboratory course will be conducted as a "local" course in contrast to a "distance" course. As such, the student is expected to attend each laboratory session from beginning to end of each session, not arrive late and not plan to leave early. Each student is required to submit Laboratory Reports individually although the experimental data used in the creation of these may be collected as a group.

Laboratory Notebook: Each student will create and maintain a Laboratory Notebook throughout this Laboratory course and is required to obtain a bound book for this purpose. It is best used as both a journal (or laboratory diary, if you will) and a workbook. This Laboratory Notebook will be reviewed and a grade applied to it based on the customary requirements for a laboratory notebook.

Experimental Protocol & Description: This document describes a particular experiment with a brief and succinct background explanation, experimental description and resulting data analysis description. An instance of this document exists for every experiment that will be performed in this course. All of these documents will be available in electronic form on RamCT. Obtain and study each one of these documents prior to arriving at the laboratory for the corresponding experiment. Understand the theoretical chemistry that you will be applying before you arrive at the laboratory using your personal library (you are developing your own library of important chemistry texts, aren't you?) or various web-based resources (beware of bogus resources - seek out reputable sources and don't accept everything that is "published"). You are not required to create hardcopy for this course but you may wish to do so for your own benefit; there are computers in the laboratory that can be used for viewing these documents (but not for the first time, of course). Do not use the laboratory printer for creating hardcopy of these documents.

Laboratory Experiments: There are a number of experiments that can be performed in this course; examples are provided below. Each student will perform four experiments. For some experiments, students will work individually and for others, in groups. Each student will not necessarily perform every experiment. Each experiment will occupy three weeks of laboratory time and a Laboratory Report will be submitted individually by each student and for each experiment.

Laboratory Report: A Laboratory Report is the final product of a Laboratory Experiment. Laboratory Reports will follow the standard Introduction, Materials and Methods, Results, Discussion, Acknowledgements and References (Appendices as needed and outside of the page limit) format found in professional journals. (Look up the *Analytical Chemistry* if you're not already familiar with it.) Each Report will be no more than eight (8) pages in length, with a separate title page and in single column format. The Laboratory Report will be submitted both (1) in hardcopy form and (2) coincidently as a single PDF file (no other format is acceptable and **a zero grade will be issued if either is not submitted**) at the beginning of the laboratory period one week after the corresponding experiment commenced. No late submissions will be accepted. (Don't Even Ask.) No "re-grades" will be performed. Plan your work accordingly. Make requests for help to the GTAs and Laboratory Instructor in a timely manner. Print your hardcopy version from the PDF version so that they are identical.

A professional style for the names of files submitted is required. Use lower case exclusively. Use the following format for Laboratory Reports and for Oral Presentations submitted in this course, respectively:

"*431_last_name_report_#1.pdf*"
"*431_last_name_oral_presentation.pdf*"

Oral Presentation/Examination: Each student will make a formal oral presentation of one of the Laboratory Experiments and Laboratory Reports and be questioned about it. The choice of the Experiment to be used in the Oral Presentation will be made by the student in consultation with and with the approval of the Laboratory Instructor. A projector and companion computer will be set up in a classroom for presentations and a laser pointer will be provided. Students will bring their presentations on their own USB disks in **PDF format only** (no other format will be accepted). Students will make a presentation in their regularly scheduled section. Each student will be allocated fifteen (15) minutes in which to make a presentation and answer questions. The presentation order will be reverse alphabetical (using the last name) and all students are expected to be present for all presentations. Students will be expected to make a serious oral presentation at an upper division university level on a topic of chemistry and to demonstrate an understanding of the material by responding to questions from the audience. Students are strongly advised to review their PDF files and practice their presentations prior to making them in class where they will be assessed and graded.

Laboratory Grades: As described in the main syllabus for this course, for each student a final laboratory grade will be combined (25%) with a final lecture grade (75%) to yield a final grade (100%). Additionally, each student must achieve a "passing" laboratory grade in order for that student to pass the entire course.

Each student's final course grade will be based on (1) the scores earned on the four Laboratory Reports (each worth 100 points), (2) the score earned on the Oral Presentation (worth 100 points) and (3) the score earned on the Laboratory Notebook (worth 100 points). The final course grade will be based on the number of points earned out of a possible 600 points. The final letter grade issued will be based on breakpoints set by the Laboratory Instructor in the final earned point distribution.

The Oral Presentation must be made and the slides must be submitted (as a PDF file) coincident with the delivery of the presentation. Failure to complete both of these tasks at the appointed time will result in a failing course grade, that is, "F."

If a student fails to submit one of the four Laboratory Reports, the Oral Presentation score will be substituted for the missing Laboratory Report score as well as being counted as the Oral Presentation score.

If a student submits all four Laboratory Reports and the Oral Presentation score is higher than the lowest of the four Laboratory Report scores, the Oral Presentation score will be substituted for the lowest of the Laboratory Report scores as well as being counted as the Oral Presentation score.

No extra credit or alternate grading schemes will be offered or issued. There will be no written examinations, including a final examination, in this laboratory course.

Laboratory Experiments: The following is a list of titles of experiments that are available in this laboratory course. The order in which these experiments are performed may vary depending upon the needs of the course. The exact schedule will be clearly communicated during the semester.

1. Measurement of the decay time of the fluorescence of aqueous quinine
2. Gas chromatography (GC) of natural products
3. Quantitative atomic absorption spectroscopy
4. Simultaneous UV-Vis spectroscopy and potentiometry on an oscillating reaction
5. Electrochemistry in aqueous solution (cyclic voltammetry)
6. High pressure liquid chromatography (HPLC) of analgesic compounds
7. Transmission and attenuated total reflectance (ATR) infrared spectroscopy
8. Determination of the pKa of a weak acid
9. Determination of reaction kinetics

Laboratory Safety: The physical safety in the laboratory of all personnel including students, GTAs and faculty is of paramount importance. Proper laboratory dress and conduct are required at all times. Students are required to secure and use their own approved (!) protective eyewear. There are no "loaners" available in the laboratory. Additionally, students are required to be suitably attired in the laboratory according to the clearly and previously identified in the lower division laboratory courses at all times. Lastly, professional and courteous conduct is required at all time. Students failing to abide

by any of these requirements will be summarily ejected from the laboratory without provision for making up any experimental work missed and without any special consideration in the evaluation and grading of the corresponding submitted materials.

University Activity Conflicts: Any student having a conflict with the Schedule described in this syllabus as the result of scheduled University Activities must to inform the Instructor via e-mail during the first week of the semester. The options will be reviewed at that time and accommodations will be made, both as provided for by University Policy. Otherwise, students are expected to be executing according to the Schedule presented in this Syllabus.

Laboratory Schedule		
Week Beginning	Topic	Notes
15 Jan	Brief Course Introduction Experiment #1	
22 Jan	Experiment #1	
29 Jan	Experiment #1	
5 Feb	Experiment #2	Experiment #1 Report Due
12 Feb	Experiment #2	
19 Feb	Experiment #2	
26 Feb	Experiment #3	Experiment #2 Report Due
4 Mar	Experiment #3	
11 Mar	University Holiday	
18 Mar	Experiment #3	
25 Mar	Experiment #4	Experiment #3 Report Due
1 Apr	Experiment #4	
8 Apr	Experiment #4	
15 Apr	Presentation on Presentations	Experiment #4 Report Due
22 Apr	Review of Draft Slides Laboratory Notebook Evaluation	Draft Presentation Slides Due (individually & in person)
29 Apr	Oral Presentation/Examination Student Course Survey	Oral Presentation File Due
6 May	Final Examination Week	No final examination for Laboratory

Topics of High Importance: The following items represent various methods and techniques that are central to the ways of Experimental Physical Chemistry.

1. Ways to prevent the contamination of solid and liquid reagents, *etc*.
2. How to properly measure static and dynamic electrical parameters.
3. How to perform high-quality, quantitative and well-controlled instrument measurements.
4. How to "build" an analytic instrument.
5. How to modify an analytic instrument.
6. How to make a recalcitrant analytic instrument submit to the experimenter's will.
7. How to read an analog instrument properly and report the uncertainty of the measurement.
8. How to read a digital instrument properly and report the uncertainty of the measurement.
9. How to create a high-quality graph and how to embed it in a document.
10. How to create a high-quality laboratory report modeled on peer-reviewed journal articles.
11. How to create robust and portable documents and presentations.
12. How to pull (wrangle? wrestle?) experimental data from scientific instruments.