

Instrumental Analysis

CHEM 4430 K

Spring 2018

Time / Date / Location

Class will meet Monday and Wednesday from 11:00 AM to 12:15 AM with a lab from 9:00 AM to 12:00 PM on Friday.

Purpose

This laboratory course is designed to familiarize the student with the physical basis and applications of chemical measurements and instrumentation. Activities will be based on electronics, optical spectroscopy, thermodynamics, kinetics and computational chemistry. This course has been designated as a writing intensive course for the *Discipline Specific Writing* (DSW) and will include writing informal and formal laboratory reports and a term paper or presentation.

Text (*Optional*) "Fundamentals of Analytical Chemistry," by Skoog, West, Holler, and Crouch. 9th Ed., Thomson-Brooks/ Cole Publisher.

Learning Outcomes

- To develop an appreciation for errors and measurements, and how to live with them.
- To learn basic electronics.
- To understand the construction of simple and complex chemical instrumentation.
- To learn how to answer both quantitative and qualitative chemical questions.
- To develop a working understanding of as wide a range of instrumental tools as possible.
- To be introduced to the analytical concepts and applications of electrochemistry, spectroscopy, and chromatography.
- Design and perform simple experiments (spectroscopy, thermodynamics, kinetics and electronics) to study chemical systems
- Operate independently and interpret results using common chemical instrumentation and modeling.
- Develop a positive attitude towards research and laboratory experimentation, which often involves unexpected difficulties in reaching research goals; and
- Communicate scientific ideas effectively in oral and written presentations

Instructors	Office	Office Hours
Douglas A. Stuart, Ph.D.	TLC 2125 678-839-6022 office 773-330-1392 cell dstuart@westga.edu	Drop-ins are usually ok, Check the door if I am out.

Exams and Quizzes

There will be a midterm and a final exam. The final exam will consist of writing and oral sections. In the oral exams students will be interviewed by the instructor to determine the extent (depth and breadth) of the knowledge they should have acquired by that point in the course. The exams will be cumulative and cover both lecture and laboratory material. The number of students will likely necessitate scheduling

some exams outwith the regular class period. Quizzes will be given weekly, usually Friday, to provide regular assessment.

Lecture

The lecture portion of the course will provide you an overview of modern chemical instrumentation. You will learn about the fundamental principles behind the machines that get the data the chemists use every day. Even those who are primarily interested in synthetic chemistry will benefit from understanding the machines they rely on to analyze their products. We hope to remove the idea that instrumentation is a “mystery box” that simply spits out results. We will learn about the inner workings of the instrumentation to understand the “why” and “how” of its operation, which can provide insight into the aptness of a given technique, if that particular machine is functioning correctly, and how to interpret the results, and troubleshoot potential problems. The lecture is also designed to directly complement the laboratory component of the course, not only to prepare you for impending activities, but also to go amplify on those aspects we will be unable to explore in lab.

Laboratory

This course is envisioned as being very “hands-on” in its approach, where learning by doing serves as the most long lasting and practical method of instruction. As such, lab will be of the utmost importance. The laboratory will have its own syllabus. It will contribute **50%** toward the final grade in the course.

Homework

Homework will be assigned, collected, and checked for completeness. Your homework must be stapled and legible when turned in.

Writing Lab Reports

One of the purposes of an advanced laboratory course is to provide experience in writing scientific reports. In accordance with the DSW requirement, each report will be edited and graded. Resubmission of your report may occur if deemed it necessary. Please pay close attention to avoiding calculation errors, since these can ruin the most carefully gathered data. Please visit the library and look at articles from a current issue of any ACS journal, e.g. *Analytical Chemistry*, to get an idea of the writing style used. To the end of your report, staple photocopies of your lab notebook pages (including calculations) which contain your data and instructor initials. **Please see the sections below on laboratory notebooks and laboratory reports.**

Policy on Late Laboratory Reports

All laboratory reports are due one week after the experiment is completed. A 10% per day penalty will be assessed for each day the report is late. If the report is not turned in by the fifth late day, the laboratory report will not be accepted. It is our policy to administratively withdraw a student from this course with a WF if they fail to turn in more than two laboratory reports within the stipulated time.

Laboratory Notebook

A laboratory notebook should provide a permanent record of details in procedure, raw data, observations, calculations and results. The criterion for sound record keeping is that someone else should be able to readily locate and understand the pertinent results for an experiment. Although reasonable legibility and neatness are required to meet this criterion, the utility of a record is determined largely by whether it is

original, systematic, and complete, not by whether it is a work of art. The following are guidelines for keeping a proper experimental record:

1. A bound notebook is required for this lab. Numbered pages are required.
2. Enter all data in **ink** as soon as it is taken. Never recopy numbers or use loose sheets of paper. Cancel errors or rejected data by drawing a single line through them. Do not erase or remove pages. Set aside the first two pages of the notebook for a table of contents, which you will organize at the end of the course.
3. Enter data only on the right page of the two facing pages (*optional*). Use the left side as a scratch sheet for calculations.
4. Clearly label all entries, including the units (*e.g.* grams). To facilitate direct recording of experimental work, it is helpful to set up a data page for each experiment before coming to the lab.
5. Even if the calculation of final results from the data taken in lab is done at a later time, record these calculations in your notebook for future reference. To avoid errors in the calculations, write out each step of the calculation.
6. Draw and sketch as you go along. Show yourself where the sample goes, what knobs or controls are present, and where the power switches are. Use the graph paper for guides, but don't worry about exact rendering. The drawings you make may become very useful later.
7. Give good descriptive names for data, preferably incorporating the date. Copied data often has the date copied, not the date the data were collected.

Report Format

All reports will be on 8.5 × 11-inch sheets and should be written on a word processor. Students are encouraged to make use of spreadsheet and plotting software in their reports. They are encouraged to plot data as soon as it is obtained, if possible, so that problems may be detected early and not the night before the write-up is due. Sloppy reports will not be graded and will be returned to be rewritten. Correct use of English is expected in all reports. If in doubt about spelling, look the word(s) up in a dictionary or run a spell checker. Be specific and concise in reports, especially in the introduction and discussion. Do not put reports in fancy binders or folders. Use a single staple in the upper left-hand corner of the report. *You are to submit your lab report as a .DOC file over CourseDen!* Students today have ready access to scanners, word processors, and email. Any lengthy mathematical calculations may be submitted as a neat recopying from the lab notebook.

Formal Reports

The student will be graded using the following format:

1. **Title:** Title of experiment, together with your name, department, and college should appear on a separate title page which is not counted in the 10-page limit.
2. **Abstract:** A one-paragraph (five to 10 sentences) abstract must be included on the lower half of the title page. The purpose of the experiment should be stated and the results summarized.
3. **Introduction:** Outline the theory of the experiment; use schematics when possible. The line of reasoning upon which the experiment is based must be presented in a careful and logical manner.
4. **Experimental:** A brief but descriptive outline or statement of the procedure should be included. Emphasize any changes of the procedure prescribed in the experiment handout. A neat sketch of the apparatus is useful. Label all parts of the drawing.
5. **Data:** Organize the data carefully into tables and/or graphs where appropriate. Include with every numerical entry an estimate of its uncertainty and the units. When several determinations are made of the

same quantity, the standard deviation of the average should be used to indicate uncertainty. A spreadsheet and plotting program **must be used** for the graphs.

6. **Calculations and Results:** One example of each calculation should be given. Each result should be accompanied by an estimate of its uncertainty (standard deviation). For replicate determinations, the final value of each sample can be calculated, the results averaged and the standard deviation can be calculated. Alternatively, an uncertainty may be estimated by propagating the uncertainties with the raw data. Learning to estimate errors and see how they propagate through the calculations into the final result is one of the most important aspects of this laboratory experience. For linear data, use least-squares analysis when appropriate.

7. **Discussion with error analysis:** Discuss how the results correlate to the theory presented in the introduction. From the statistical analysis data, discuss the significance of the results obtained. Consider whether the precision of the results is reasonable for the particular method of analysis. Describe any potential sources of determinate or systematic error.

Informal Reports

Informal reports must not exceed 5 total pages.

1. **Title:** Title of experiment, together with your name, department, and college should appear on a separate title page which is not counted in the 5-page limit.

2. **Abstract:** A one-paragraph abstract must be included on the lower half of the title page. In the abstract, you must briefly state the purpose of the experiment and summarize your findings. This paragraph should be limited to no more than five sentences.

3. **Data:** Organize the data carefully into tables and/or graphs where appropriate. Include with every numerical entry an estimate of its uncertainty and the units.). For replicate determinations, the final value of each sample can be calculated, the results averaged and the standard deviation can be calculated. You must use a spreadsheet and plotting program for the graphs. **No hand-drawn graphs will be accepted.**

4. **Calculations and Results:** One example of each calculation should be given. Deviation of experimental results from theoretical data should be presented. A detailed error analysis is **not** required for these reports.

5. **Discussion:** Discuss how the results correlate to the theory presented in the introduction.

In-class Reports

In-class reports must not exceed 2 total pages.

1. **Title:** Title of experiment, together with your name, department, and college should appear on a separate title page which is not counted in the 2-page limit.

2. **Abstract:** A one-paragraph abstract must be included on the lower half of the title page. In the abstract, you must briefly state the purpose of the experiment and summarize your findings. This paragraph should be limited to no more than five sentences.

3. **Data:** Organize the data carefully into tables and/or graphs where appropriate. Include with every numerical entry an estimate of its uncertainty and the units. For replicate determinations, the final value of each sample can be calculated, the results averaged and the standard deviation can be calculated. You must use a spreadsheet and plotting program for the graphs. **No hand-drawn graphs will be accepted.**

4. **Calculations and Results:** One example of each calculation should be given.

Term Paper and Poster on an Instrumental Method

Students will select an instrument or instrumental method of interest of them, to be approved by the instructor. At the end of the semester, the students will write a 10-page, double spaced, font size 12, 1

inch margins term paper in which they summarize the instrument or method. The paper should discuss the scope, theory and impact/application of the techniques used. In addition, each student will present a “research poster” presenting their topic .

The assignment on the term paper will be broken into four parts:

- I. **Initial Draft** – submit a 5-page (excluding figures) draft of your paper for editing. **Due on March 28, 2018.**
- II. **Final Paper** – submit the 5 to 10-page final draft of your paper along with a copy of your initial draft. **Due on April 18, 2018.**
- III. **Poster Presentations will be held on the last day of class**

Attendance Policy

You are expected to be present and prepared for each class meeting. Due dates for homework and examinations are scheduled and will not be moved. No late work will be accepted and no make-up exams will be given.

E-mail Policy

The primary means of communication with the instructor will be via e-mail. Since email has proliferated, and now constitutes the bulk of extra-classroom conversation between student and instructor, it must be subject to normal rules of formality. Therefore, all e-mail communication will follow the guidelines enumerated here. E-mail should be composed in formal, professional language, and with attention to the propriety accorded to the position of the writer, and the addressee. E-mails that do not meet these standards will not be returned by the instructor. E-mail should not ask questions whose answers are contained in the course syllabus. Such e-mail will not be returned by the instructor. Students should avoid asking questions in e-mail that should be raised either in class, or in individual consultation with the instructor. These include questions of an excessively conceptual nature, and questions that expect an unreasonable amount from the instructor. A good rule of thumb: if you question cannot be answered in two sentences or less, or if it is a question that you should solve on your own through the course of your reading, then it is not appropriate for e-mail.

Lastly, e-mail will only be answered during normal work hours (9am – 5pm) Monday through Friday. E-mails sent outside of those hours, or on the weekends, *will not* be returned until the resumption of normal business hours.

Academic Dishonesty

The Honor Code of the University of West Georgia is in effect. Any infractions will be mediated through this process.

Grades

Your course average will be calculated as follows:

$(\text{Exam Average}/\text{Quiz Average} \times 0.4) + (\text{H.W.} \times 0.1) + (\text{Laboratory Average} \times 0.5) = \text{Course Average}$

[90% - 100% = A] [80% - 89% = B] [70% - 79% = C] [60% - 69% = D] [0% - 59% = F]

Tentative Schedule

Due to instrument availability, we will likely work in small groups. Even in small groups, there is not enough equipment for each team to have exclusive access to a given instrument each week. We will circumvent this difficulty by developing a rotation. Thus by mid February, the lecture and the specific lab you will perform may diverge. We will use the lecture session on Friday to play “catch up” for those who will out of synchronization with the lecture presentations.

Week	Subject
1	Introduction, Error Analysis, Lab Records
2	Analog Electronics (voltage divider, low pass filter)
3	Analog Electronics
4	Analog Electronics
5	Digital Electronics (labview student edition, thermal probe)
6	Digital Electronics
7	Vacuum Equipment (integrate press sensor with DAQ)
8	Spectroscopy: UV Vis
9	Spectroscopy: Raman, Mass Spec, InfraRed MIDTERM
10	Separations: GCMS
11	Separations: Electrophoresis
12	<i>Spring Break – Classes</i>
13	Microscopy SEM, XRD
14	Microscopy AFM
15	Electrochemistry: Ion sensitive electrodes
16	NMR
16.5	Oral Exam

SAFETY RULES: INSTRUMENTAL ANALYSIS

As a student, I will adhere to the following safety rules:

1. I will follow all general and specific instructions given by the instructor for a lab activity.
2. I will study my assignment. If I am in doubt about any procedure, I will ask the instructor or one of the laboratory assistants for help.
3. I will not perform activities that are unauthorized, nor will I engage in inappropriate behavior while I am in the laboratory.
4. I will dress sensibly and appropriately for lab. I will avoid long, loose sleeves, dangling ear rings and other large items of jewelry, and clothing that interferes with movement. I will not work in the laboratory with bare feet, open-toed shoes, or high heels.
5. I will wear protective eyewear (safety glasses or goggles) throughout the entire period that I am in the laboratory.
6. I will use the safety equipment provided when needed. I will become familiar with the locations of the fire extinguisher, the safety shower, the eye wash, and the first aid kit.
7. I will immediately report to the instructor any accident, injury, incorrect procedure, or damaged equipment.
8. I will place broken glass and waste chemicals in designated containers. I will not dispose of any hazardous chemical down the sink. If I am not sure of how to dispose of any chemical, I **WILL ASK MY SUPERVISOR!**
9. I will not bring into the laboratory any food, drink, chewing gum, or tobacco products, except for what may be stored in book bags.
10. I will follow good housekeeping and good safety procedures in cleaning spills, replacing caps on containers, and replacing materials that are for general use.
11. I will wash my hands thoroughly immediately after leaving the laboratory.
12. I will report the loss or breakage of any materials, or any damage of any instrument immediately.

STUDENT AGREEMENT: INSTRUMENTAL ANALYSIS

Please acknowledge that you are prepared to start class by initialing the following statements and signing the document below. This will help to ensure that we are all aware of the basic operation for advanced instruction in chemistry.

<u>Statement</u>	<u>Initial</u>
1. I have read and understood the safety rules above	_____
2. I know how to calculate my grade	_____
3. I am aware of the policy on late submission of lab reports, and the requirement that they be submitted electronically.	_____
4. I know the exams will have written and oral components	_____
5. I am aware of the e-mail policy	_____
6. I have read the syllabus and know the due dates for my paper, and the exam schedule	_____
7. I keep a neat and complete lab notebook	_____

Printed Name

Signature

Date