

Chemistry 414: Instrumental Analysis
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Office hours by appointment or just drop by

Chemistry 414 is a hands-on technical lab. You will be expected to read the background material associated with each technique in your text and download and read each experiment before coming to lab. Once in the lab, you will prep the samples for each experiment and collect data from the instrument being used in the experiment. Once you have gained experience with the different instrumentation, you will identify a problem that can be solved using one of the pieces of instrumentation. You will research the background and instrumental methods used and perform the experiments needed to determine the answers to your questions. Finally, you will write a formal report and present your findings in front of the class. This guide contains information that may answer many of questions that arise during the semester. A copy of this guide will be available through the Sakai resources tab of the course.

Lab Policies

Missed Labs

Your semester grade will be based on completing and writing up all laboratory experiments. None of these labs will be dropped. Ideally you will be able to attend all your labs, but makeup labs will be offered periodically through the semester. Coordinate making up any missed labs with your TA.

Outstanding Bills

Bills must be paid by the first reading day after classes end. If you have an outstanding bill at that time you will have a hold put on your account. Keep this in mind if you need to have your grades on record for a scholarship or GRADUATION!

Safety Equipment

You need to have safety glasses, gloves and a lab coat for the lab. If you do not have safety equipment or are wearing improper footwear, you will be asked to leave the lab and may not return until properly dressed. You can purchase the safety equipment in the stockroom, Room 210, but you must bring your ID card with money on a ram account since they don't accept cash.

Safety eyewear is available through the graduate students outside Beaupre 115 at the start of the semester. After that they are available through the stockroom. **We are unable to loan either glasses or goggles due to RI state health laws, so please do not ask if you can borrow safety glasses.**

If you have long hair, you must tie it back during lab. We also recommend long pants rather than shorts or skirts in case of spills.

Lab Notebook

You must purchase a carbon copy lab note book. They are available in the bookstore. All observations and data taken during your experiment must be recorded in this notebook. Use a black or blue pen, pencil is not allowed. As a means of checking your data recording skills, you will be required to turn in the carbon copy of the data with each experiment, so make your notes clear and concise. A calculator will be required during many of the labs. You are not allowed to use a cell phone as a calculator.

Written Procedures

There is no lab manual associated with the course. All lab procedures must be downloaded from the Resources section of Sakai. Bring a copy of the procedure to each lab. The experiments will be posted at least 1 week before you will do each lab. Some of the experiments require you to do prelab questions, so please check the lab early. Download the lab early in case any problems arise. You must also bring a copy of the experiment to each lab. You are not allowed to use a tablet or phone for reading the procedures during lab. No excuses will be accepted for not getting a copy of your experiment before you come to lab.

Late Policy

All work must be handed in within 15 minutes of the start of the lab session. Work done on both Tuesday and Thursday of one week is due at the start of the lab session the following Tuesday. If the work has not been completed it can be handed in Thursday of that same week for a 10-point penalty. If handed in a full week late (the following Tuesday), 20 points will be deducted. No work will be accepted more than 1 week late under any circumstances.

Teaching Assistant

The teaching assistant for the course is Satu Heiskanen. Please contact her using by email if you want to set up an appointment to review any of the material in the course or get help with calculations. Her office hours are by appointment on Mondays and Wednesdays. Her email is sheiskanen@chm.uri.edu. If you cannot make either of those days, please email her to arrange another time.

Satu will give a brief overview of the theory and instrumentation being used in the lab at the start of each experiment. You will be expected to read up on each technique on your own before the experiment starts. You will be expected to know the general theory, instrumentation, applied mathematical calculations and the advantages/disadvantages of all the instruments presented in the course for the final, so take notes early.

Lab Grading and Point Distribution

Satu is responsible for all the lab grading. See her immediately if you have a problem with the grading in your lab. If the problem does not get resolved, then contact me.

Grading for Lab:

All teaching assistants will be grading according to the following point distribution.

| | | |
|----------------|-----------------|-------|
| Short reports | 100 points each | 50% |
| Lab notebook | 100 points | 10% |
| Project report | 100 points | 20% |
| Presentation | 100 points | 10% |
| Written final | 100 points | 10% |
| | | <hr/> |
| | | 100% |

Short Lab reports

A short lab report is expected for each experiment performed. The rubric for grading each lab report will be included in the downloaded experiment. **Your original data sheets are very important and must be submitted with the short report.** Each person must use their own data when performing calculations. Unless told otherwise, you will work individually, not in groups. If you submit the same data, graphs, etc. as another person in the course, you will be given a zero for that portion of the experiment. You will also be graded on the quality of your lab work, so don't rush and be sure to perform each step of the experiment carefully. You will be expected to read up on each technique on your own, since only a brief overview of each technique will be given in lab. You will be graded on your preparedness for each experiment, so be sure to understand the basic concepts before your lab session.

Formal Lab Report and Presentation:

One formal lab report will be written based on the special project selected. A presentation based on this report will be given at the end of the semester. The specific requirements for the special project will be discussed in February.

Lab Notebook

A carbon copy lab notebook is expected to be used in the lab at all times. Carbon copies of the data/observation pages must be passed with each lab report. Black or blue ink is required for full credit. Expectations of what is required in the lab notebook will be discussed in more detail during check-in.

Written Final

A written final will be given during the regularly schedule finals session. The assigned time slot for this year's final will be **Thursday, May 4th from 8-11am**. The final will cover the theory and practice of each of the instruments used in the course. Be sure to read each section of the text to fully understand the background of the material. Notes will also be posted in Sakai that can help you better understand each piece of equipment.

Tentative Lab Schedule

| Week | Dates | Day | Tentative 414 Schedule |
|------|-----------|-------------------------|--|
| 1 | 1/23-1/27 | M Tu W Th F | Check in and lab introduction Introduction to spectroscopy |
| 2 | 1/30-2/3 | M Tu W Th F | UV Spectroscopic Analysis of Caffeine & Benzoic Acid in Soft Drinks Simultaneous Determination Of Chromium And Cobalt by Visible Spectroscopy |
| 3 | 2/6-2/10 | M Tu W Th F | Atomic Absorption Determination of Zinc & Copper in a Multivitamin Atomic Emission Determination of Sodium in Tap Water |
| 4 | 2/13-2/17 | M Tu W Th F | Fluorescence Spectroscopy: Introduction and prep Determination of Fluorescein in Antifreeze by Fluorescence Spectroscopy |
| 5 | 2/20-2/24 | M Tu W Th F | Ion Selective Electrodes: Determination of Fluoride in Toothpaste GC with FID detection: Determination of Alcohols by Gas Chromatography |
| 6 | 2/27-3/3 | M Tu W Th F | HPLC with diode array detection: Introduction and prep Reverse Phase HPLC: Quantitative Analysis of Polyaromatic hydrocarbons |
| 7 | 3/6-3/10 | M Tu W Th F | HPLC with fluorescence detection: Introduction and prep HPLC with fluorescence detection: analysis |
| 8 | 3/13-3/17 | | Spring Break |

| Week | Dates | Day | Tentative 414 Schedule |
|------|-----------|-------------------------|--|
| 9 | 3/20-3/24 | M Tu W Th F | GC-MS: Introduction and prep GC-MS Analysis of an Unknown Mixture |
| 10 | 3/27-3/31 | M Tu W Th F | LC-MS: Introduction and prep LC-MS Analysis |
| 11 | 4/3-4/7 | M Tu W Th F | Projects Projects |
| 12 | 4/10-4/14 | M Tu W Th F | Projects Projects |
| 13 | 4/17-4/21 | M Tu W Th F | Projects Projects |
| 14 | 4/24-4/28 | M Tu W Th F | Presentations Final papers due in lab |
| 15 | 5/1-5/5 | M Tu W Th F | Last day of classes Reading day Reading day Final: 8am-11am |

Example Grading Rubrics: More specific rubrics will be included with each experiment

Short Reports

Lab reports are to be typed. Full sentences are to be used at all times. Include the following sections in the following order:

| | | | | | |
|-------|--|-------|---|-------|-----------------------------|
| _____ | Title Page (5pts) | _____ | Course and Title of Experiment | | |
| | | _____ | Name of Researcher | | |
| | | _____ | Date of experiment | | |
| _____ | Instrumentation and Procedures (1page maximum including drawing) (10 pts) | _____ | Chemical theory behind instrumentation | | |
| | | _____ | Drawing and description of each part of the instrument (if copied, cite source) | | |
| | | _____ | Advantages/Disadvantages of this type of instrumentation | | |
| | | _____ | Common applications of this type of instrumentation | | |
| | | _____ | Brief description of experiment and why it is advantageous to use this instrumentation for this particular analysis | | |
| _____ | Calculations and Questions (50pts) | _____ | Data correlates to original data sheet (no partial credit if they do not match) | | |
| | | _____ | Calculations and answers to questions are correct | | |
| _____ | Tabulated data and Results (10pts) | _____ | <i>typed tables with all</i> data and results (correct significant figures and units required) | | |
| | | _____ | graphs as required | | |
| | | _____ | in the same order as calculations | | |
| | | _____ | data transcribed correctly from data sheet and sample calculations | | |
| | | _____ | correlates to original data sheet and calculations | | |
| _____ | Conclusions (15pts) | _____ | No more than ½ page | | |
| | | _____ | Clear and accurate summary of experimental results | | |
| | | _____ | 1-2 sentences supporting/refuting success of experiment (percent error, etc.) | | |
| | | _____ | Clear final sentence about success in relation to purpose of experiment | | |
| _____ | Lab Technique (10points) | _____ | no disruption of lab | _____ | clean & organized work area |
| | | _____ | proper waste disposal | _____ | proper safety procedures |
| | | _____ | copy of experiment present | | |
| _____ | Total Score | | | | |

Lab Notebook

The following are required for each experiment. Pass in the carbon copy with each short report.

Validation

- _____ Title of Experiment
- _____ Name of Researcher
- _____ Date of experiment
- _____ Signed by TA as a witness to the experiment
- _____ Page number(s) (if not preprinted on page)

Procedures: (Done as you go along with the experiment, not written ahead of time, it must include what you did in the lab

- _____ Model number and type of equipment used must be recorded
- _____ Specific information (eg. 100mL Class A volumetric flask, chemical mass or volumes, etc.) must be included with all procedures recorded
- _____ You must use full sentences
- _____ All entries are written in black or blue ink
- _____ Corrections must be made with single line cross-out, initialed and dated

Data Records

- _____ Significant figures and tolerances on data are included and based on equipment used
- _____ Units are included on all data
- _____ Observations are recorded on separate lines from data
- _____ All entries are written in black or blue ink
- _____ Corrections must be made with single line cross-out, initialed and dated

Your lab notebook will be 20% of your grade. You will be graded with a separate lab notebook grade for each experiment. It will count for 1 lab notebook grade. This will give you feedback on recording laboratory data correctly.

After all labs are complete, you will need to pass in a carbon copy of the following information. This information will be collected at the end of the semester and checked for completeness.

Title page

Include a title page in your lab note book with your name, the course, section, time and instructor and date of first entry. Add date of last entry when book is closed out at the end of the semester.

Table of contents

Leave the next 2 pages free for a table of contents. After each lab is completed, record the first page number of each experiment to create your table of contents.