Chemistry 3210 Laboratory
Instrumental Analysis - Laboratory
Spring 2021

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Course Description: This is a second lab course in analytical chemistry for Chemistry majors and other advanced students needing a Chemistry core. The course serves as a laboratory which accompanies the corequisite lecture course CHM 3210 (Instrumental Methods of Analysis). The purpose of this lab class is to provide students with a hands-on overview of modern chemical instrumentation: what the devices are, how they work, what design features are important, and applications to chemical problems. Students will obtain experience with instruments representing the major areas of spectroscopy and chromatography.

Course Objectives: The student should complete this course having obtained an overview of modern instrumental analytical chemistry. The student should be familiar with pragmatic calibration and operation of modern chemical instrumentation. The student should be capable of selecting (and then implementing in practice) appropriate techniques for solving specific problems in analytical chemistry, and should have an understanding of pertinent interferences, limitations, quality assurance practices, and procedures for evaluating the accuracy and precision of the resulting data.

Course Structure/Approach: The class will be conducted primarily in a laboratory setting. Students will work in cooperative groups of 2 students (group sizes will be adjusted according to enrollment). Students will receive some formal instruction from the instructor and/or assistant prior to commencing an experiment, but will largely work independently thereafter. The instructor and assistants will be available during the scheduled laboratory time. ***Students should note that laboratory work may need to be conducted outside of the normal lab periods as required by logistics. These times will be set up in consultation with the laboratory assistant and/or Instructor.***

Textbook and Required Materials: Experiment handouts as provided on the Canvas **Lecture** page; Harris will be useful as a reference book.

Electronic Resources: Electronic copies of all lab-related course materials will be available on

Canvas. Normally, distribution of printed material will be minimal. Please see CHM 3210 lecture syllabus for more discussion of electronic resources and web-based journal articles.

Course Policy Statement, Evaluation Methods, and Deadlines

*** You will work in small groups** as a matter of logistics. All lab reports are to be prepared and submitted individually, and will be graded as such. Should a series of lab reports written by group members display a blatantly strong degree of non-individual effort, the group will be warned and penalized; further occurrences will be considered as violations of academic integrity.

*** Corrected lab reports are not to be shared** with any students who have not yet turned in the lab. This will be considered a violation of academic integrity.

*** Practices strictly prohibited** include: fabrication of data and writing a report using what is obviously someone else's data (unless approved by the instructors due to exceptional circumstances). These two practices will be considered to be violations of academic integrity.

*** Lab data** are to be recorded in a carbon-type notebook, with carbon pages turned in with the report. If you do not have a carbon-type notebook, then you will need to turn in photocopies/pictures with your lab report (do NOT turn in .jpeg, .heic, or any other picture files. Please convert to a .pdf using an app such as CamScanner). An existing notebook with adequate space may be used. No work may be chicken-scratched on paper towel, scrap paper, toilet paper, etc. Under all circumstances, all group members should maintain adequate individual notebooks which reflect efforts of lab partners as required. For example, if a needed reagent is prepared by one group member, all group members should note its preparation and acknowledge who prepared it. Hard-copy output from instruments will be re-printed or photocopied so that each group member has a complete set of documentation. Photocopying (as needed) of laboratory output may be done in the Chemistry Department office with consent of the TA or instructor.

*** Attendance is required: Failure to complete even 1 laboratory experiment will result in a zero (0) for the laboratory section of the course.**

*** Makeup labs:** Makeups of labwork missed on these dates will be done at an agreeable time provided a reasonable excuse exists for the absence. Note that one member of a group who misses one or both dates of a group experiment will most likely make up the entire experiment as a solo effort. The instructor is reasonable but reserves the right to deny makeup lab time for frivolous excuses.

*** There is not a perfect relationship** between the timeframe of coverage of specific topics in 3210 lecture and the 3210 laboratory. In fact, the relationship is almost non-existent. Therefore, you are expected to be reasonably familiar with the topic of the present experiment through your own reading.

*** There will be no examinations, but there will be an oral quiz with the Teaching Assistant.** Your grade will be assigned based principally upon your lab reports. There are 300 possible points as follows:

Four short lab reports (Stats, ICPMS, HPLC, GC/MS, UV/VIS): 50 points each	250 points
Oral Quiz on one of the above experiments	50 points
Instructors' evaluation of technique, cleanliness, effort, and effectiveness in a team environment	50 points

*** The format for the lab reports** will be clarified with the handouts. Basically, you will be expected to provide your data along with some derived results, sample calculations, and answers to some specific questions. **In all reports, please be sure to state the origin of any uncertainty assumptions/measurements**. The text portion of all lab reports should be typewritten, preferably using a computer word processor, and submitted as hardcopy. As is most pragmatic for you, you may submit requested calculations as handwritten attachments, as typewritten material, or as printout from computer computational software packages (e.g. EXCEL, etc.). It is recommended that you retain a copy of all submitted materials for your records. Each experiment can be completed in two weeks and **the report (or oral quiz) is due 1 week afterwards.**

*** In lieu of a written lab report, one oral quiz will be taken with the TA.** You should be prepared to discuss, in detail, the theory behind the instrument, questions regarding your specific data, and everything in between. The goal is to be able to talk, in real time, about your grasp of the data, as well as the instrument which produced this data.

*** Late lab reports** will possibly be assessed a penalty of 5 % of the maximum possible grade per week late. **Persons submitting lab reports after the same report has been returned to others shall submit an affidavit stating that the writer has not examined any graded reports of other students**. No late lab reports will be accepted after 5:00 PM on Tuesday, Jun 8, 2021.

*** You are welcome to use any Departmental or DU computer facilities** in preparing your lab reports, or any of your own resources. Users of DU or Departmental computer facilities are required to abide by the adopted policies and procedures.

*** Some experiments will involve the quantitative analysis of an appropriate unknown.** In most cases, emphasis will be placed upon whether your answer is "correct" with respect to the data you obtained. Many unknowns are genuinely "unknown", for which the instructor and TA do not have an absolute answer. In one case, the unknown is a standard reference material for which there will be a performance-based grade component.

***** The most important point to make about **safety in the laboratory** is that safe work habits are the most important protection in the lab. If you adopt safe work habits, and learn proper technique, the chemical laboratory is much safer than the typical home or athletic facility. Were you to be asked to work in an instructional chemical laboratory with anything as dangerous as, for example, home drain cleaners or paint remover, we would provide extensive cautions, and provide selected protective wear.

Accidents need not happen if you are careful, and are constantly aware of what you and your neighbors in the lab are doing, and why. Use common sense, and above all, think! Responsible behavior is essential. No horseplay in the lab. Dress safely, act safely. The following comments highlight specific safety aspects of laboratory work in this course.

1. Think about what you are doing. Plan ahead. Do not "cookbook" – it is neither safe nor effective learning. If you give no thought to what you are doing, you predispose yourself to an accident.

2. Carefully read the directions for each laboratory experiment or measurement before coming to the laboratory. Answer questions in the "pre-lab" before coming to the laboratory. An unprepared student is a hazard to everyone in the room.

3. Wear approved eye protection at all times in the laboratory and in any area where chemicals and glassware are handled. The only exception is when explicit instructions to the contrary are given by your instructor. For example, the time in the laboratory might be divided up such that for some period everyone was working on computers, and no chemicals and glassware were being handled, and the instructor might then suggest that you could work without wearing safety glasses.
    a. Eye protection should protect against impact and chemical splashes. Wrap-around safety glasses are safer than standard eye-wear. Goggles are strongly recommended and may be required, if the instructor judges that eye protection is inadequate.
    b. Safety experts are of divided opinion about the safety of wearing contact lenses in the laboratory. The contact lens may provide extra protection against a direct insult, but on the other hand they may prevent adequate flushing of the eye in case of an accident, and some vapors could dissolve in the lenses or get behind the lens. If you normally wear contact lenses, please ask for additional background information regarding safety so that you can make your own judgment in consultation with your physician. If you decide to wear contact lenses in the lab under regular safety glasses, be sure those working near you, and your instructor, know that you are wearing contact lenses.
    c. If you should get a chemical, or a particle of something, in your eye, was with flowing water from the eyewash fountain for at least 15 minutes. The immediately get medical attention.

4. Fashions change with time. Some years ago we would not have had to include guidance such as "don't wear roller blades in the lab"! Now, this is a real safety issue. Similarly, long hair and short clothing and gaps in clothing are current issues. Any layer of clothing is an additional layer of protection for your skin. Long hair, dangling jewelry, etc. can get into reagents and transfer chemicals to you, or minimally, mess up your experiment. Confine long hair. If any skin other than your hands is bare, including when you are reaching, wear a lab coat or apron. Wear sensible shoes in the laboratory. Sandals are prohibited, because of dangers from broken glass, dropped objects, or chemical spills.

5. Perform no unauthorized experiments. This includes using only the materials and quantities instructed, and no more.

6. Consult your instructor if you have any doubts about the instructions in the laboratory manual, or if you feel that any operation you are about to perform is unsafe or beyond your physical abilities. No operation in a laboratory should require excessive force. If you are uncertain, or uneasy, about some manipulation in the laboratory, ask for help. If you are not confident that you can perform an experiment safely, don't do it.

7. Do not eat, drink, or smoke in the laboratory at any time. Do not put pens or other items in your mouth. The obvious hazard is the transfer to your mouth of materials that could injure you.

8. Be sure to read labels on chemical containers carefully.

9. Be careful to avoid contaminating reagents. Never return reagents to the bottle. Close containers after use.

10. Do not touch chemicals with your bare hands. Some chemicals are absorbed through the skin. Wash off all chemicals with large quantities of running water.

11. Avoid breathing fumes of any kind.
    a. To test the smell of a vapor, collect some in a cupped hand and bring it toward your nose cautiously.
    b. Never smell a chemical reaction while it is occurring.
    c. Work in a hood if there is the possibility that noxious or poisonous vapors may be produced.

12. Never use mouth suction in filling pipets with chemical reagents. Always use a pipet bulb.

13. A clean and neat work area is a safer work area. Clutter not only will slow your work, but it will lead to accidents. Another good work habit is somewhat analogous to "defensive driving" – that is, always assume that someone else will do something that could injure you. Consequently, wear your safety glasses at all time, not just when you are doing something that would require their use.

14. Never work alone in the laboratory. There must always be at least one other person present in the same room. In addition, an instructor must be quickly available.

15. In case of fire or accident, call the instructor at once.

16. If you spill something, clean it up quickly, even if it is "only water." Ask for help if you cannot safely put down what is in your hands at the moment. Wet floors are a common occurrence, and are very dangerous.

17. If you spill acid or base on yourself, immediately wash it extensively. Then, apply some of the concentrated $NaHCO_3$ solution that is provided for this purpose.

18. Report all injuries to your instructor immediately. Except for very superficial injuries, you will be required to get medical treatment for cuts, burns, or fume inhalation. Your instructor will arrange for transportation if needed.

19. We are also concerned with safety of the environment. Please dispose of chemicals in the containers provided (and labeled).

20. No chemicals, glassware, or equipment are to be removed from the lab.

21. Clean up your work space, including wiping down the surface and putting away all chemicals and equipment, at the end of the lab period.

22. Wash your hands with soap and water before leaving the laboratory.

*** Grading Guidelines:** The key elements of the report will include a cover page that summarizes your findings, completed worksheet, and all appended data requested in the laboratory instructions. Each laboratory will be worth 50 points total. Completion of pre-lab requirements will be worth 5 points; laboratory reports will be worth 45 points. Reports will be graded in accordance with the following breakdown:

A. Cover page/Memo (15 points)

- Your name and names of all lab partners that participated.
- The date the lab was completed and the date it was turned in.
- The name of your Lab TA
- Concise description of the purpose of assigned lab and instrument used. Do not copy and paste from the lab statement – this should be your own interpretation.
- Concise summary of your findings, including results (**with uncertainty reported**). The summary should be **only 2 paragraphs**. Here, you should be able to succinctly summarize i) what you did, ii) pertinent results, and iii) why these results are meaningful. The ability to summarize large amounts of data is an acquired skill, and you will be graded on how effectively you can achieve this. As Mark Twain said,

  "*I'm sorry this letter is so long, I didn't have time to make it shorter.*"

  Long-windedness is not rewarded in Chem 3210, or any of the sciences! See any abstract in a scientific article for examples.
- Remember to watch your significant figures!
- Spelling and grammar **are** important.
- Format – 12 point Times New Roman font; one inch margins, single-spaced.
- Maximum length – 1 page.

B. Worksheet (20 points)

- Address the questions put forth in the specific lab statements
- Specific model numbers of instruments, including manufacturer, column type, etc.
- Summary of **all** sample calculations –
  - **Do not assume that your TA or Instructor knows where a value came from**. If we cannot **quickly** figure out a value's origin, then we will assume you made it up.
- Appropriate error included with all your reported values (summary of calculations of error NOT required here IF they are documented in the appended data. NOTE: **you must tell us on the worksheet were we can find the calculations if you choose this approach**).
- References cited where appropriate (**there should be some with all labs**). You will have to search the literature to find relevant articles.
- Results of any QA / QC work. This may include, but is not limited to:
  - Statistical analysis and results from reference materials (SRMs)
  - Blank analysis over the course of the run
  - Instrumental drift analysis (rerun calibration standards)

C. Required data (10 points)

- A photocopy (or carbon copy) of all lab notebook pages containing raw data and calculations.
- A copy of all required chromatograms or spectra asked for in lab instructions.
- **Tables summarizing collected data and calculated values.**
- Figures of calibration curves.
- Copy of Excel spreadsheets when regression analysis (LINEST) is required (UV/VIS).

Schedule for Spring 2021: CHEM 3210 Lab
Note that weeks go from Wed - Tues

GROUP	A	B	C	D
Week 1 (March 31 – Apr 6)	Check in / Statistics			
Week 2-3 (Apr 7 – 20)	ICPMS	GCMS	UV-vis	HPLC
Week 4-5 (Apr 21 – May 4)	GCMS	UV-vis	HPLC	ICPMS
Week 6-7 (May 5 – May 18)	UV-vis	HPLC	ICPMS	GCMS
Week 8-9 (May 19 – Jun 1)	HPLC	ICPMS	GCMS	UV - vis
Week 10 (Jun 2 – 8)	Make–up / check out			

Be sure that you schedule an oral quiz with the appropriate TA.