# Chemistry 3210 Laboratory
# Virtual Instrumental Analysis - Laboratory
# Spring 2020


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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:** By necessity, this course will be conducted virtually. The goal of the laboratory section of the class is to give the full laboratory experience, without actually controlling the instrumentation. This is, of course, somewhat contradictory. In the experiments where data analysis is the primary learning objective, and instrument control is secondary (UV-vis and ICP-MS), you will effectively perform the entire experiment (pre-lab and data work-up), but the TA will have collected the data for you. In this case, the report expectations have not significantly changed. In other experiments where instrumentation controls are primary and data anlaysis secondary (e.g., GCMS), the experiments are based on online instrument simulations. In no way will this take the place of actually sitting at the instrument, but there are actually some decent and more than acceptable simulations out there. The instructor and assistants will be available during the scheduled laboratory time.

**Textbook and Required Materials:** Experiment handouts as provided; Harris will be useful as a reference book.

**Electronic Resources:** Electronic copies of all lab-related course materials will be available on Canvas. 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 individually, since there is no consideration of physical limits.** All lab reports are to be prepared and submitted individually, and will be graded as such. Should a series of lab reports display a blatantly strong degree of non-individual effort, the writers 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 truly exceptional circumstances). These two practices will be considered to be violations of academic integrity.

*** Despite the unusual circumstances, lab data** are to be recorded in a carbon-type notebook, with carbon pages turned in with the report. 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, you should maintain adequate individual notebooks which reflect efforts of lab partners as required.

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

*** 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, nor a final exam.** Your grade will be assigned based principally upon your lab reports. There are 300 possible points as follows:

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| Four short lab reports (ICPMS, HPLC, GC/MS, UV/VIS): 50 points each | 200 points |
| Oral Quiz on one of the above experiments | 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 a *pdf and a *doc/*docx. 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 addition to 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 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 Friday, June 5, 2020.

*** If on campus and they are available, 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.

*** 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.
- 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 (if applicable).
- 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 where 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 (ICPMS, UV/VIS).