

Department of Chemistry CHEM 3203: Advanced Analytical Laboratory Winter 2024



Dr. Mark Wall

Lab:

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Course Description

An introduction to the fundamentals of instrumental chemical analysis is presented in a laboratory environment with emphasis on selection of appropriate analytical techniques, sample treatment, data handling, and communication of experimental results. Instrumental techniques include chromatography, spectrophotometry, mass spectrometry, and electrochemistry, with applications in biological, environmental, forensic, and health-related areas.

Prerequisites: CHEM 2201 or equivalent with a grade of C- or better.

Exclusion: CHEM 3201

Text: An Introduction to Analytical Chemistry, Doucette, A; Guy, R; Wentzell, P (A digital version of this text is offered free through Brightspace.)

Lab & Learning Outcomes

The learning outcomes associated with each experiment are described in your lab manual. There are several overarching themes:

- Time management, including proper planning and familiarization with the theory and protocols ahead of lab, and effective use of time during lab.
- Team work with lab partner, TA's, and instructors, including requesting assistance when needed, and providing assistance when possible.
- Written communication by formulating clear and concise responses within lab reports and through your posters.
- Oral communication with partner, TA's, and instructors, including describing your research project to the class.

- Effective reporting of data in graphical and tabulated formats.
- Calculation of concentrations, unit conversion, and statistical analysis.
- Problem solving related to diagnosing a problem, researching and attempting a solution, and assessing alternative solutions.
- Critical assessment of the quality of your work.
- Demonstration of proper lab technique.
- Creativity and independence.
- Working safely in the lab.
- Become familiar with the fundamental theory and practical application of the following analytical techniques:

- atomic and molecular absorbance and emission	- analytical sample preparation
– gas and liquid chromatography	– buffer preparation
- mass spectrometry	- analyte extraction
– tandem MS and different modes of ionization	- derivatization
– electrophoresis	– purification and recovery
- titrations	– methods of calibration
- ion selective electrodes	– internal standards and standard addition

Course Format

While there is no associated lecture component of the course, this class still places emphasis on fundamental understanding of the theoretical principles governing the analytical experiments being conducted in lab. Experiment 1 will be conducted during the first 4 lab periods. This experiment is a review of analytical concepts and laboratory techniques. Students are expected to know these concepts to ensure success with the remainder of the course. The practice labs include a series of videos and practice problems as a review of basic analytical concepts. Students may wish to refer to their 2nd year analytical textbook if they are not fully comfortable with these concepts. Additionally this lab aids in students knowing/understanding the expectations of pre-lab preparation, in-lab organization, hands on laboratory skills, and the expectations of CHEM 3203 reports.

Experiments 2 through 6 each constitute 2 or 3 days of lab and will be completed in groups of 2 or 3 students. Lab partners will change as the term progresses. The individual order for completing these experiments will be posted on the course website. Each experiment concludes with a written lab report (refer to section "Pre-labs & Reports" for further information).

The Experiment 7 research project will be completed over a 3 week period. This project will also be completed in groups of 2. Deadlines for associated aspects of the project (e.g., selection of topic, formal proposal, and detailed experimental outlines) will be posted throughout the term. The project concludes with a poster presentation based on the research project. Refer to section "Research Project" for more information.

There will be a written in-person final exam as scheduled during the final exam period.

Course Assessment & Dates

Assessment	Weight	Date		
Pre-labs & Reports (Experiments 1-6)	25%	As scheduled ¹		
In-lab Quizzes	10%	As scheduled ¹		
Research project proposal	2%	Feb 16		
Midterm	15%	Feb 28 (6pm-7:30pm)		
Lab performance				
Exp $1-6$	4%	Jan 8 - Mar 7		
Research project	3%	Mar 11 - Mar 28		
Progress Reports	3%	As scheduled ¹		
Poster presentation		April 3		
closed session	5%	12 3 pm		
open session	3%	35pm		
Final Exam [*]	30%	Scheduled by registrar		

¹Consult the calendar on Brightspace for due dates. *Final exam is a cumulative written test covering all aspects of the course.

Conversion to Final Letter Grades as per Dalhousie Common Grade Scale

$\mathbf{A}+$	(90 - 100)	B+	(77 - 79)	C+	(65 - 69)	D	(50 - 54)
А	(85 - 89)	В	(73 - 76)	С	(60 - 64)	\mathbf{F}	(< 50)
A-	(80 - 84)	B-	(70 - 72)	C-	(55 - 59)		

Lab Manual

The CHEM3203 lab manual will be provided digitally on Brightspace. This document contains safety information, theory, background, and experimental details as well as the pre-lab and post-lab report questions. The lab manual serves as a primary resource for information in addition to the textbook and students are required to read the relevant sections prior to attending lab and prepare their laboratory notebook with relevant information for that day.

Required Materials

The course will involve significant amounts of research and data manipulation, as such, the use of a personal laptop during lab time is recommended. Laboratory laptops will be available for use. Students must bring bring their own pens, pencils, and black permanent markers to label glassware, and a hardcover lab notebook for recording pre-lab and experimental data.

Safety glasses (CSA-Z94-3 or ANSI Z87) and knee-length lab coats are required. Both are available for purchase at the Dalhousie bookstore. More details on lab safety can be found in the section below and in your lab manual.

Lab safety

The Chemistry Department recognizes its responsibility to undertake laboratory teaching activities in a safe and environmentally responsible fashion. Responsibilities of the University, departmental chairs, laboratory supervisors, staff and students are defined in Dalhousie policy manuals made available by the Office of Environmental Health and Safety at https://www.dal.ca/dept/safety.html. A detailed description of laboratory safety is provided in your manual.

Students must complete a Workplace Hazardous Materials Information System (WHMIS) and Laboratory Safety training within the last three years. Links to register and location to upload completion certificates can be found in Brightspace. The due dates for the WHMIS course and Laboratory Safety is January 14, 2024 and January 21, 2024 respectively, by 11:59PM.

Cancellations

Weather-related closure of the University may impact submission of your lab report, in which case, the due date will be moved to the next day that the University is open. In the event of a missed lab due to school cancellation, adjustments to the class schedule and weighted value of work may occur.

Assessments

While the course has no official lecture component, there are regular assessments in the form of in-lab quizzes, a midterm, and final exam.

In-lab Quizzes

In-lab quizzes will be conducted on scheduled quiz days, see course schedule on Brightspace, and are typically on the first day of a new experiment. Quizzes will cover the learning objectives of the previous experiment. For example, on the first day of Exp 2 students will write an in-lab quiz related to experiment 1. The in-lab quizzes will typically be about 20 minutes in length.

Midterm and Final Exam

The midterm will occur during a scheduled evening time, see "Course Assessment & Dates". The date of the final exam will be scheduled by the registrar's office. The midterm, covering learning objectives in Experiments 1 - 4, and the final exam, covering learning objectives from the entire course, will evaluate your understanding of the theory, and data analysis. The exams will require the use of computers. You may bring your personal laptops for use, but the laboratory laptops will also be available. Should you miss the midterm, no makeup will occur as the data and figures will be public at that time. The grade value will be moved to the weight of your final exam.

Expectations for Lab Days

Beginning of Lab

Students arriving to lab should enter through the main doors of the analytical chemistry lab room 111P located on the first floor of the chemistry building. Personal belongings can be stored in a cubby located by the rear exit door. Lab coats and safety glasses should be worn, and pens, markers, hard cover notebook, laptops should be brought to their assigned work station. Assigned stations will be posted to Brightspace, and will change with each experiment.

At this time, experiment preparedness, in the form of pre-lab questions/information relevant to the modules being covered that day are present in their hardcover notebook. Students must be prepared properly each day attending lab for a given experiment to receive the full 1 pre-lab grade for that experiment.

A short presentation by the instructor outlining important information, background and theory, and announcements will be given. If an in-lab quiz is scheduled for that day, it will occur prior to starting any lab work.

Hardcover Notebook

Recording detailed observations is an important skill and a learning objective of this course. As you work your way through an experiment, collected data, relevant information, changes to procedures, and calculations, should all be recorded into your hardcover notebook. At any point during lab you may request assistance from the instructor or a TA, but you will likely be asked to show notes from your hardcover notebook to help them troubleshoot any problems. Pre-lab information relevant to the lab day should be written in the hardcover notebook prior to coming to lab. The date and experiment title should also be included.

Before Leaving Lab

Sharing Raw Data

Prior to leaving lab, students are required to submit a raw data sheet containing a copy of their data and information which they have recorded into their hardcover notebook. Copies of any data from instrumentation, such as Excel files, must also be copied to the analytical lab computer. Leaving without sharing raw data may result in the lab performance grade not being awarded.

Lab performance

Developing good lab habits, such as properly labelling samples and solutions, keeping a clean and neat workspace, and sharing data, is a learning objective of this course. Prior to leaving lab each day, a group's workspace will be inspected by the instructor or TA during raw data collection. Satisfactory cleanup and sharing of raw data will award the group their lab performance mark for that day. If a request by the teaching team is given to the group to improve their workspace, the request must be completed by the students prior to leaving and awarding the lab performance mark. If students leave prior to cleaning up their workspace and sharing raw data, they may not receive marks for lab performance for that lab day.

Labs end strictly at 5:30PM each day. Please budget at least 15 minutes at the end of every lab period to fill out the raw data sheet, transfer data, and clean up your work space. In many cases, unfinished work can be completed on the next lab day unless it is the last day of that experiment. Be efficient during lab and manage your time to ensure you can complete all parts of an experiment. Otherwise, you may not have the experimental data to fully complete lab report questions in the discussion section of the submitted lab report.

Pre-labs & Reports

Pre-labs

Pre-lab questions/tasks relevant to the modules being completed on the day attended, located in the CHEM3203 lab manual, must be completed and recorded in your hardcover notebook prior to conducting the experiment for that day and will be checked at the start of each lab period. It is encouraged to complete the pre-lab well in advance of the experiment and seek assistance with your teaching assistants or instructor if you encounter any confusion.

Lab Reports

Experiments 1 through 6 are graded through written lab reports. Reports are due on the listed due dates (by 11:59pm) and to be submitted via Brightspace as a single pdf file. If completed before the due date, reports may be submitted earlier. **Only digital copies in PDF format will be accepted.**

All data collected within a group for a given experiment is shared among all members of the group, regardless of the workload shared in lab. This applies even if the student was absent from that lab, see sections "Missed Labs & Illness" and "Before Leaving Lab" for further information). The report must exclusively present the

results collected within the student's own group. While collaboration and communication between lab members is encouraged, **reports must be prepared individually by each member**, and direct copying of reports in whole or in part constitutes plagiarism and will be forwarded to the Academic Integrity Officer for review.

Experiments 1 through 6 are to be graded out of 10 marks; 9 marks for the evaluation of the experiment reports and 1 mark for evaluation of pre-lab preparedness. Of the 9 marks for the report, 6 are awarded for answering the report questions given in the lab manual in the discussion section, and 3 awarded for the other sections of the report. See subsection "Lab Report Formatting" for more information. The final grade, out of 10, for all experiments will have an assigned weight percentage that will sum up to 25% of the course grade.

The percent total for each report in relation to the calculation of the final grade is presented in the table below.

Report	Total $\%$ of Final Grade
Experiment 1	3
Experiment 2	4
Experiment 3	4
Experiment 4	4
Experiment 5	4
Experiment 6	4
Experiment 6 - Presentation	2

To pass the course you must submit 5 of the 6 reports from experiments 1 through 6. All reports will be graded. For example, if you submit 5 of the 6 reports you will receive a grade of zero for the 6th report. If you only hand in 4 reports you will not be allowed to pass this course. See section "Missed Labs & Illness" for more information.

Lab Report Formatting

A template for lab reports will be provided through Brightspace. Lab reports will be comprised of sections, including Summary/Abstract, background, experimental, discussion (here is where the lab report questions found in your lab manual will be answered), as well as a conclusion. These sections will have word limits to encourage concise and efficient communication. Figures, tables, and supplementary data will not count towards the word limits for a given section.

Late report policy

One lab report during the term may be handed in up to one week late without penalty so long as the instructor is informed by email prior to the normal due date. Further late lab reports will receive a grade of zero. Illness does not grant further extension of reports (i.e., you still only get an extension for one report).

Research Project

During the last three weeks of the course, students will use their scheduled lab times to conduct a research project, see section 'Course Assessment & Dates". This project will include the submission of a research project proposal and project progress reports for review. The purpose of the research project is to give students an introduction into literature searching, experimental design, problem solving, and written/oral scientific communication. The research project will conclude with a poster presentation located in the chemistry department advanced resource centre.

Research Project Proposal

A research project proposal is required prior to starting the research project. The report will outline the background, theory, and goal of the project, required materials, chemicals, instrumentation, as well as referenced

literature and safety considerations. The proposal will be reviewed and feedback from the instructor and teaching assistants will be provided.

Progress Reports

Progress reports will be completed throughout the duration of the Experiment 7 research project. The content of the progress reports will vary week-to-week, however, each will include an updated plan for the work to be conducted during the upcoming week. The intent of these reports are to have you preparing various components of the research poster over the three weeks of research. Details of each report will be available on Brightspace and discussed within the lab. Progress reports are due at the start of each research week (see laboratory calendar). Failure to complete/submit a progress report demonstrates unpreparedness and is a safety hazard. Students will not be allowed to conduct experiments in the lab until a progress report is submitted.

Poster Presentation

The poster presentation will consist of a closed and open session. During the closed session, students will present the results of their research project to the instructor and selected faculty members and answer questions. The open session invites all members of the chemistry department and possibility other members of the faculty of science to view the posters and ask questions of the students.

Missed Labs & Illness

All missed labs require a declaration of illness. If a student misses a single lab day they must contact the instructor via email as soon as possible. The student will not be given opportunity to make up the missed lab day, but the absent student is still required to submit a lab report on the normal due date. Data collected by the group will be shared with the absent student, and their pre-lab grade for the experiment will be based on their preparation for the remaining lab days attended for that experiment.

Essays

One Day Missed

To substitute for a single missed lab day, the absent student is also expected to **submit a 1000–1500 word** essay due together with the report. The essay topic is chosen by the instructor and relates to the material covered in the given experiment. This essay will be graded on a scale of 0 to 3 (0 = not completed; 1 = partially complete, or poorly done, essentially a failing grade; 2 = a minimal effort, essentially a grade of C- or below; 3 = acceptable). The corresponding percentage will be multiplied by the submitted report grade, then pre-lab grade applied based on preparation for the attend lab sessions.

For example, a student misses Exp 2 Day 1 and writes an essay and submits it along with the Experiment 2 lab report prior to the due date. They receive a grade of 2/3 for their essay and a 8/10 for their report (note: prelab mark is applied at the end of the calculation). The grade for their lab report would be $8/10 \times 2/3 = 5.3/10$. The student was well prepared for the days they did attend lab and were awarded the full 1 pre-lab mark. The pre-lab grade is now applied for a final grade of 5.3/10 + 1/10 = 6.3/10 for the experiment.

More than One Day Missed

If more than one day is missed for a given experiment the data collected by the group will no longer be shared with the absent partner and the remaining partner will be offered assistance in lab to complete the experiment.

To substitute for a lab report for that experiment, the absent student will be required to write **three 1000–1500** word essays due at the start of the final exam covering topics related to the learning objectives of the experiment, as chosen by the instructor. The essays will again be graded on a scale of 0 to 3. The averaged grade percentage of the three reports will be applied by the final exam grade to determine the grade for the

report.

A reminder that students must complete a minimum of 5 out of the 6 lab reports. The three essays do not substitute for a report. Thus, students cannot miss more than one experiment. Exceptional circumstances due to extended illness will be considered for alternative grading schemes.

Accommodation Policy for Students

Students may request accommodation as a result of barriers related to disability, religious obligation, or any characteristic protected under Canadian Human Rights legislation. Student Accommodation Policy: (www.dal.ca/access).

Students who require accommodation for classroom participation or the writing of tests and exams should make their request to Academic Support prior to or at the outset of the regular academic year. More information and the Request for Accommodation form are available at www.dal.ca/access.

Academic Integrity

Academic integrity, with its embodied values, is seen as a foundation of Dalhousie University. It is the responsibility of all students to be familiar with behaviours and practices associated with academic integrity. Instructors are required to forward any suspected cases of plagiarism or other forms of academic cheating to the Academic Integrity Officer for their Faculty.

Policy on Intellectual Honesty and Faculty Discipline Process

All students are expected to follow the Dalhousie policies on intellectual honestly. Each class you take, exam you write and assignment you complete will have different rules depending on the instructor. But no matter what you are required to do in order to earn your degree, Dalhousie University expects all students to be responsible learners, which means that you will complete assignments yourself and acknowledge sources of information and ideas when they are not your own, among other things.

Use of Generative AI

Students in this course are expected to develop their own writing skills and style. The submission of any work, such as lab reports, research proposal, or essays, created through the use of generative AI tools, (e.g., ChatGPT, Bard, Midjourney, etc.), is considered a breach of student academic integrity and intellectual honesty and all suspected cases will be brought forward to the Academic Integrity Officer. The use of generative AI to assist in the aid of studying and understanding of theory and background material is permitted, although effort should be made to request URL links to resources as information generated through these tools can be factually incorrect.

Student Code of Conduct

Dalhousie University has a student code of conduct, and it is expected that students will adhere to the code during their participation in lectures and other activities associated with this course. http://www.dal.ca/dept/university_secretariat/policies/student-life/code-of-student-conduct.html.

Copyright

All members of the Dalhousie community are expected to comply with their obligations under Canadian copyright law. Dalhousie copyright policies and guidelines, including our Fair Dealing Guidelines, are available at https://libraries.dal.ca/services/copyright-office.html.

Diversity and Inclusion – Culture of Respect

Every person at Dalhousie has a right to be respected and safe. We believe inclusiveness is fundamental to education. We stand for equality. Dalhousie is strengthened in our diversity. We are a respectful and inclusive community. We are committed to being a place where everyone feels welcome and supported, which is why our Strategic Direction prioritizes fostering a culture of diversity and inclusiveness Statement: http://www.dal.ca/cultureofrespect.html

Recognition of Mi'kmaq Territory

Dalhousie University would like to acknowledge that the University is on Traditional Mi'kmaq Territory. The Elders in Residence program provides students with access to First Nations elders for guidance, counsel and support. Visit or e-mail the Indigenous Student Centre (1321 Edward St) (elders@dal.ca). Information: https://www.dal.ca/campus_life/communities/indigenous.html.

Student Resources and Support

University Policies and Programs

Important Dates in the Academic Year (including add/drop dates):
http://www.dal.ca/academics/important_dates.html

Classroom Recording Protocol: https://www.dal.ca/dept/university_secretariat/policies/academic/classroom-recording-protocol. html

Dalhousie Grading Practices Policies: https://www.dal.ca/dept/university_secretariat/policies/academic/grading-practices-policy.html

Grade Appeal Process:

https://www.dal.ca/campus_life/academic-support/grades-and-student-records/appealing-a-grade.
html

Sexualized Violence Policy: https://www.dal.ca/dept/university_secretariat/policies/health-and-safety/sexualized-violence-polic html

Scent-Free Program: https://www.dal.ca/dept/safety/programs-services/occupational-safety/scent-free.html

Learning and Support Resources

General Academic Support - Advising (Halifax): https://www.dal.ca/campus_life/academic-support/advising.html

Student Health Wellness Centre: https://www.dal.ca/campus_life/health-and-wellness.html

On Track (helps you transition into university, and supports you through your first year at Dalhousie and beyond): https://www.dal.ca/campus_life/academic-support/On-track.html Indigenous Student Centre: https://www.dal.ca/campus_life/communities/indigenous.html

Indigenous Connection: https://www.dal.ca/about-dal/indigenous-connection.html

Elders-in-Residence (The Elders in Residence program provides students with access to First Nations elders for guidance, counsel, and support. Visit the office in the Indigenous Student Centre or contact the program at elders@dal.ca or 902-494-6803: https://cdn.dal.ca/content/dam/dalhousie/pdf/academics/UG/indigenous-studies/Elder-Protocol-July2018.pdf

Black Student Advising Centre: https://www.dal.ca/campus_life/communities/black-student-advising.html

International Centre: https://www.dal.ca/campus_life/international-centre.html

South House Sexual and Gender Resource Centre: https://southhousehalifax.ca/about/

LGBTQ2SIA+ Collaborative: https://www.dal.ca/dept/vpei/edia/education/community-specific-spaces/LGBTQ2SIA-collaborative. html

Dalhousie Libraries: http://libraries.dal.ca/

Copyright Office: https://libraries.dal.ca/services/copyright-office.html

Dalhousie Student Advocacy Services: https://www.dsu.ca/dsas?rq=student%20advocacy

Dalhousie Ombudsperson: https://www.dal.ca/campus_life/safety-respect/student-rights-and-responsibilities/where-to-get-help ombudsperson.html

Human Rights and Equity Services: https://www.dal.ca/dept/hres.html

Writing Centre: https://www.dal.ca/campus_life/academic-support/writing-and-study-skills.html

Study Skills/Tutoring: http://www.dal.ca/campus_life/academic-support/study-skills-and-tutoring.html

Faculty of Science Advising Support: https://www.dal.ca/faculty/science/current-students/undergrad-students/degree-planning.html

Safety

Biosafety: http://www.dal.ca/dept/safety/programs-services/biosafety.html

Chemical Safety:

https://www.dal.ca/dept/safety/programs-services/chemical-safety.html

Radiation Safety:

http://www.dal.ca/dept/safety/programs-services/radiation-safety.html

Laser Safety:

https://www.dal.ca/dept/safety/programs-services/radiation-safety/laser-safety.html