



Experimental Chemistry II (Integrated Chemistry Laboratory)

COURSE INSTRUCTOR:

Prof. Igor Kozin

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OFFICE HOURS: Mondays and Wednesdays, 1:30 to 2:30 PM (starting from week 3 of classes).

COURSE DESCRIPTION and GOALS

CHEM 397 F/W (CHEM 398 F, plus CHEM/ENCH 399 W) is a laboratory course introducing modern experimental methods in synthetic organic, inorganic, and biological chemistry, physical and computational chemistry, material science and analytical chemistry. Integration of several experimental methods is emphasized in the synthesis, chromatographic separation, and spectroscopic characterization of various chemical species. This course offers the students a unique opportunity to gain valuable hands-on skills working with scientific instruments and techniques commonly used in many academic and industrial laboratories.

INTENDED STUDENT LEARNING OUTCOMES

Upon completion of this course, the students will be able to:

- Develop critical skills necessary for the analysis of original experimental data by applying established theoretical concepts in Chemistry and related disciplines
- Formulate scientific reports based on the critical evaluation of available theoretical and experimental data
- Perform complex lab procedures independently or with little supervision to implement various chemical reactions in organic/inorganic synthesis and catalysis
- Perform complex chemical separation and purification steps for subsequent compound characterization studies
- Perform compound and material characterization studies using common spectroscopic techniques, such as ^1H - and ^{13}C -NMR, ATR- and DRIFT-IR, UV-VIS and fluorescence spectroscopy
- Perform qualitative and quantitative chemical analysis using instrumental detection techniques based on gas- and liquid chromatography
- Using specialized software, e.g.: Gaussian 03, apply basic methods in computational chemistry to evaluate fundamental chemical and physical properties of chemical compounds
- Perform extensive literature studies using online resources to identify and get access to published information pertaining to various fields in fundamental and applied Chemistry, and related disciplines

COURSE WEBSITE

Students registered in this course can access the **OnQ** course space online from: <https://onq.queensu.ca/>.

The course site contains the following information:

- Course syllabus, required and recommended materials, tutorial and final exam information
- Your grades
- Lab-related information, including safety, administration and report requirements, the lab schedule, detailed marking outlines, lab videos, Lab TA notes
- TA contact information

TUTORIAL:

One-hour tutorial sessions will be conducted weekly either in-person (room CHE 117) or, if announced, as MS Teams remote meetings; attendance of all tutorials is mandatory. Quizzes on studied tutorial material will be conducted either as 'take-home' exercises or as in-person tests.

NOTE: No final examination will be held. To pass this course, each conducted tutorial quiz must be passed, i.e.: your marks for each quiz must be greater than 50%.

LABORATORY: Two three-hour lab sessions weekly as scheduled (*Chernoff Hall, room 120 – 122 and NMR facility*).

CHEM 397 students will complete 18 two- or three-session lab experiments; CHEM 398 and CHEM 399/ENCH 399 students will complete 9 labs in each term, see LAB INFORMATION below for more details.

All components of this course will receive numerical percentage marks. The final grade will be derived by converting your numerical course average to a letter grade according to the Queen's official grade conversion scale (see below).

COURSE GRADING SCHEME

Laboratory (18 reports for CHEM 397, 9 reports for CHEM/ENCH 398/399): **70%**

Tutorial Quizzes (5 quizzes in total, including the '*Spectroscopic Identification of an Unknown*' quiz): **30%**

Your course grade will be based on your individual marks received for all completed labs and quizzes.

Depending on the lab complexity and duration (two- and three session labs), individual lab marks will range from 5 to 7% of the total lab grade (70%).

The tutorial component will include '*take-home*' exercises on practical aspects of using scientific literature resources and interpretation of spectroscopic data (10.5% of the total grade for four quizzes) and the '*Spectroscopic Identification of an Unknown*' quiz (19.5% of the total grade), which will be held during the last scheduled lab session).

To pass this course, you must pass each lab and tutorial quiz.

REQUIRED MATERIALS

- Approved, lab coats and lab goggles. Depending on the Queen's COVID-19 policies, you may be asked to attend the labs while wearing non-surgical face masks and face shields, instead of lab goggles.
- Chem397/398/399 lab manual

Calculator Policy

- As noted in Academic Regulation 9.2, Calculators acceptable for use during quizzes, tests and examinations are intended to support the basic calculating functions required by most Arts and Science courses. For this purpose, the only approved and permitted calculator for Arts and Science courses and exams is the **Cassio 991 series**.
- RECOMMENDED TEXTBOOKS:
 - Fessenden, Fessenden and Feist. "*Organic Laboratory Techniques*"
 - Pavia, Lampman, Kriz, 5th ed. (or more recent) "*Introduction to Spectroscopy*" or R.M. Silverstein '*Spectrometric Identification of Organic Compounds*', 7th ed
 - Atkins and De Paula. Physical Chemistry (10th or 11th ed)
 - Carey and Juliano. Organic Chemistry (9th or 10th ed.)
 - Harris. Quantitative Chemical Analysis (8th or 9th ed)
- RECOMMENDED SOFTWARE:
 - *MarvinSketch* (*chemaxon.com*, free download, request a free license from course instructor)
 - Bruker *Topspin* (*bruker.com*, obtain free academic license) or *MestReNova* (*mestrelab.com*, free download but will require a license)
 - MS Excel and Word, and OneDrive (provided by Queen's)

Lab Notebook - carbonless copies are a must – you will hand those copies to your TAs.

Grade	Numerical Course Average (Range)
A+	90-100
A	85-89
A-	80-84
B+	77-79
B	73-76
B-	70-72
C+	67-69
C	63-66
C-	60-62
D+	57-59
D	53-56
D-	50-52
F	49 and below

LAB FORMAT, ATTENDANCE and LAB EVALUATION POLICY

1. CHEM 397, CHEM 398 and CHEM/ENCH 399 lab experiments will be carried out in-person in pre-assigned lab groups, according to a 'rotating' lab schedule printed in this Laboratory Manual and posted on the course OnQ site. Your experiment schedule will be defined by your lab group number, which you should obtain during the Introductory session for your specific lab section (see the lab schedule below). Students must attend all labs scheduled for their lab group. If a lab is missed due to extenuating circumstances, the student may be able to perform the missed work with a different lab section (conditional to the equipment and TA supervision availability). Permission of the course instructor is required for any lab work with the alternate lab section. Please inform the TA in your lab section and course instructor as soon as possible about any expected problems with lab attendance and scheduling.
2. Each 3rd year lab experiment may take **two or three lab sessions** to complete, as scheduled. You will be expected to work on all aspects of lab experiments in your initially assigned lab group for the duration of this course. If, for any reason, you are unable to collaborate productively with your lab group partner(s), you may be asked to complete your lab reports individually.
3. **Prelabs and completed lab reports** must be uploaded on the course onQ site using the experiment-specific Assignment folders. **Lab reports must be submitted within one week after completing the last lab session of each specific experiment.** However, if you experience time/assignment conflicts with different courses, you can submit your report within a 3-days grace period without penalty. Longer accommodations would require an authorization from the Queen's Accommodations Office and must be discussed with the course instructor as soon as possible. Generally, the TAs are expected to grade your lab report within one week after its submission in onQ. Each lab grade will include the Lab Performance component (to be assigned by the TAs at their discretion). The TAs will enter lab grades in the Queen's OnQ database (<https://onq.queensu.ca/>). You are strongly encouraged to discuss any problems with respect to reports grading or regarding any other aspects of the laboratory evaluation first directly with your TAs and then with the course instructor. Note that lab grades assigned by the TAs are provisional, and the course instructor will finalize all grades at the end of the laboratory period in each term.
4. Acceptable quality lab reports (greater than 50% of the lab grade) will be required for all completed lab experiments scheduled for your lab group. If you fail your first lab report, you will be able to re-write it for a passing grade. **Subsequently failed lab reports will result in the overall failing grade of the entire course.**
5. You are expected to have read the procedures and to understand the reactions and apparatus before starting an experiment. Before you begin an experiment, obtain the TA's approval. Normally, this approval will be signified by the TA initialing the page in your laboratory notebook that contains the title of the experiment.
6. If you finish a part of your scheduled lab experiment ahead of time, or in cases where an experimental procedure calls for allowing a reaction to reflux or stand until the next laboratory period, ask your TA for permission to use the time to proceed with the lab further, or to catch up with unfinished earlier experiments (e.g. spectroscopy).
7. This class offers the student a unique opportunity to work with instruments that are commonly used in many research and industrial laboratories. This equipment is very sensitive and not necessarily designed for the multiple-user mode necessary for this class. The instruments are prone to breakdown and expensive to repair. We ask, therefore, that everyone takes good care of the instruments. Cleaning the spills of chemical in the vicinity of the equipment and the instrument itself is especially important. Consult your TA if unsure about the proper cleaning technique for a particular instrument (e.g.: an analytical balance, a sample compartment of a spectrometer, etc.) You will be graded on lab techniques, which involves making sure that the instrument is in the proper condition before, during and after its use. If complaints occur from subsequent groups, you may lose part or all of your lab performance grades.

8. Laboratory Notebooks

Relevant notes should be written for each lab are to be kept in a bound, ruled notebook that makes carbonless copies. You must write a Table of Contents, and all entries in the book are to be made in ink. Handwriting must be neat and legible. Data are to be entered in the notebook at the time they are obtained. None of the original pages should ever be torn out of the notebook, but you will be required to submit the carbonless copies as part of your lab reports. Each page should also give the date the work was done.

9. Lab Performance Grades

Each lab grade will also include the *Lab Performance* component (to be assigned by the TAs at their discretion). When evaluating your Lab Performance, the TAs will consider the following questions:

- ✓ Were the students well prepared for the lab in terms of their understanding of theoretical concepts and experimental techniques employed in the lab?
- ✓ Did the students implement all lab procedures (e.g.: weighing, pipetting, preparing samples/solutions, conducting instrumental measurements, lab cleanup, etc.) with the required care and attention to detail, and hence recorded adequate data?
- ✓ If your experimental data are poor and you struggled doing the lab, e.g.: mishandling samples, cross-contaminating pure solvents and reagents, etc., your lab performance marks could be deducted up to 100%. However, if your lab has been compromised by instrumental problems unrelated to your conduct (e.g.: software malfunctioning, etc.), your performance marks will not be affected.

Any lab accidents should be reported immediately to the TA or Lab Technologist. Your TAs are authorized to deduct points from your laboratory mark for safety violations or for hiding or failing to report an accident or spill. However, you will NOT be penalized for reporting accidents or spills, even if they caused the problem.

ACADEMIC INTEGRITY

Queen's students, faculty, administrators, and staff all have responsibilities for upholding the fundamental values of academic integrity; honesty, trust, fairness, respect, responsibility and courage (see www.academicintegrity.org). These values are central to the building, nurturing and sustaining of an academic community in which all members of the community will thrive. Adherence to the values expressed through academic integrity forms a foundation for the "freedom of inquiry and exchange of ideas" essential to the intellectual life of the University (see the Senate Report on Principles and Priorities <http://www.queensu.ca/secretariat/policies/senate/report-principles-and-priorities>).

Students are responsible for familiarizing themselves with the regulations concerning academic integrity and for ensuring that their assignments and their behaviour conform to the principles of academic integrity. Information on academic integrity is available in the Arts and Science Calendar (see Academic Regulation 1 <http://www.queensu.ca/artsci/academic-calendars/regulations/academic-regulations/regulation-1>), on the Arts and Science website (see <https://www.queensu.ca/artsci/students-at-queens/academic-integrity>), and from the instructor of this course. Departures from academic integrity include plagiarism, use of unauthorized materials, facilitation, forgery and falsification, and are antithetical to the development of an academic community at Queen's. Given the seriousness of these matters, actions which contravene the regulation on academic integrity carry sanctions that can range from a warning or the loss of grades on an assignment to the failure of a course to a requirement to withdraw from the university.

Copyright of Course Materials

Course materials created by the course instructor, including all slides, presentations, handouts, tests, exams, and other similar course materials, are the intellectual property of the instructor. It is a departure from academic integrity to distribute, publicly post, sell or otherwise disseminate an instructor's course materials or to provide an instructor's course materials to anyone else for distribution, posting, sale or other means of dissemination, without the instructor's express consent. A student who engages in such conduct may be subject to penalty for a

departure from academic integrity and may also face adverse legal consequences for infringement of intellectual property rights.

Accommodations with Disabilities

Queen's University is committed to achieving full accessibility for people with disabilities. Part of this commitment includes arranging academic accommodations for students with disabilities to ensure they have an equitable opportunity to participate in all of their academic activities. The Senate Policy for Accommodations for Students with Disabilities was approved at Senate in November 2016 (see <https://www.queensu.ca/secretariat/sites/webpublish.queensu.ca.uslclwww/files/files/policies/senateandtrustees/ACADACCOMMPOLICY2016.pdf>). If you are a student with a disability and think you may need academic accommodations, you are strongly encouraged to contact the **Queen's Student Accessibility Services (QSAS)** and register as early as possible. For more information, including important deadlines, please visit the QSAS website at: <http://www.queensu.ca/studentwellness/accessibility-services/>

Academic Consideration for Students in Extenuating Circumstances

Queen's University is committed to providing academic consideration to students experiencing extenuating circumstances that are beyond their control and are interfering with their ability to complete academic requirements related to a course for a short period of time. The Senate Policy on Academic Consideration for Students in Extenuating Circumstances is available at:

<http://www.queensu.ca/secretariat/sites/webpublish.queensu.ca.uslclwww/files/files/policies/senateandtrustees/Academic%20Considerations%20for%20Extenuating%20Circumstances%20Policy%20Final.pdf>

Each Faculty has developed a protocol to provide a consistent and equitable approach in dealing with requests for academic consideration for students facing extenuating circumstances. Arts and Science undergraduate students can find the Faculty of Arts and Science protocol and the portal where a request can be submitted at: <http://www.queensu.ca/artsci/accommodations>. Students in other Faculties and Schools who are enrolled in this course should refer to the protocol for their home Faculty.

Because of the course complexity and logistical constraints, the students should discuss any academic considerations for this course directly with the course instructor at the earliest possible opportunity.

If you need to request academic consideration for this course, you will be required to provide the name and email address of the instructor/coordinator. Please use the following:

Instructor/Coordinator Name: **Prof. Igor Kozin**

Instructor/Coordinator email address: *igor.kozin@chem.queensu.ca*

Acknowledgement of Territory

Queen's University is situated on traditional [Anishinaabe and Haudenosaunee Territory](#).

Discussion Guidelines

University is a place to share, question and challenge ideas. Each student brings a different lived experience from which to draw upon. To help one another learn the most we can from this experience please consider the following guidelines.

1. Make a personal commitment to learn about, understand, and support your peers.
2. Assume the best of others and expect the best of them.
3. Acknowledge the impact of oppression on the lives of other people and make sure your writing is respectful and inclusive.
4. Recognize and value the experiences, abilities, and knowledge each person brings.
5. Pay close attention to what your peers write before you respond. Think through and re-read your writings before you post or send them to others.
6. It's ok to disagree with ideas, but do not make personal attacks.

7. Be open to being challenged or confronted on your ideas and to challenging others with the intent of facilitating growth. Do not demean or embarrass others.
8. Encourage others to develop and share their ideas.