Course Instructor: Associate Professor Ignacio Vargas-Baca  
Office: ABB-265  
Ext.: 23497

Graduate Teaching Assistants: Alex Hudson, Chris Franko, Lucia Lee, Peter Ho.

E-mail: Please use the mail function of Avenue to Learn to contact the instructor and teaching assistants for all issues pertaining to this course, other than self-reporting of absences (see below).

Technical Administrators: Leah Allan (ABB-113; x22486; allanle@mcmaster.ca)  
Karen Neumann (ABB-412; x26084; neumann@mcmaster.ca)

Course webpage: on McMaster’s Avenue to Learn: The laboratory manual is available as a series of pdf files on the course Avenue site.

Course Subject: Advanced techniques for synthesis and characterization of organic and inorganic molecules and materials, and the use of modern instrumentation in chemistry.

Prerequisites: CHEM 2LA3 and registration in an Honours Chemistry program.


Required Course Materials:  
Hardbound “Physics Notes” laboratory notebook  
Safety goggles  
Lab coat

Course Objectives. Chemistry 2LB3 and is a continuation of its 1st-term partner course, Chemistry 2LA3. The primary goal of these courses is to train students in the fundamental techniques of modern experimental chemistry: the synthesis and spectroscopic characterization of organic and inorganic molecules and materials, chemical analysis, the measurement of physical properties of molecules and materials, and computational chemistry. Secondary goals include training in proper record-keeping and safe laboratory practices, and the development of critical thinking and time-management skills. The specific experiments to be carried out in the course have been designed to give students experience in the fundamental aspects of both molecular and materials chemistry, thus providing them with a sound basis for selecting their individual area of specialization in Levels 3 and 4 of the Honours Chemistry program, and preparing them for the Level 3 laboratory course, where more advanced techniques will be learned and there is a greater emphasis on inquiry. An emphasis has been placed on the design of integrated laboratory experiments, that emphasize the interplay between the traditional sub-disciplines of analytical, inorganic, organic, and physical chemistry.

Course Structure. While the course instructor is ultimately responsible for Chemistry 2LB3, day-to-day instruction and supervision, and weekly evaluation of laboratory notebooks will be provided by a team of graduate teaching assistants, with technical support from undergraduate laboratory staff members. Both the course instructor and the teaching assistants contribute to student evaluation in the courses.
Evaluation

<table>
<thead>
<tr>
<th>Component</th>
<th>Percentage</th>
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<tbody>
<tr>
<td>Pre-lab preparation, including lab book and pre-lab quizzes</td>
<td>15%</td>
</tr>
<tr>
<td>Laboratory notebook &amp; short reports of laboratory results</td>
<td>30%</td>
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<tr>
<td>Skills Assessment</td>
<td>15%</td>
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<tr>
<td>Poster Presentation</td>
<td>10%</td>
</tr>
<tr>
<td>Formal Lab Reports</td>
<td>30%</td>
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Grades for pre-lab preparation will be assigned continuously throughout the course, and on an experiment-by-experiment basis. Each experiment in the laboratory manual provides explicit instructions in regards to pre-lab preparation, which takes two forms in every case:

**Mandatory readings.** The description of the experiment itself must be read from start to finish before entering the laboratory.

**Pre-lab Quizzes:** Students will be given ~15 min pre-lab on-line quizzes.

**Notebook preparation.** The “Pre-lab” section of each experiment lists specific things that are to be entered into the notebook prior to entering the laboratory for that experiment. These requirements have been standardized for the labs in 2LB3. Pre-lab content will be graded by the TAs during the experiment, according to the following standards:

- **Excellent:** student has read the experiment and essential readings, the notebook has been prepared as instructed, and the student is clearly well-organized.
- **Good:** student has read and understands the experiment and the essential readings and prepared the notebook as instructed, but there are minor deficiencies.
- **Poor:** student has read the experiment and the essential readings and prepared the notebook as instructed, but there are major deficiencies in the notebook preparation.
- **Please See Instructor:** student is unprepared for the experiment; without notebook entries and/or reading of the lab manual.

Students who are unprepared for the experiment will be required to withdraw from the laboratory for the day, and make an appointment with the course instructor, who will decide whether the circumstances warrant allowing the lab to be made up on an alternate date. If a make-up is permitted, it will be the student’s responsibility to arrange a date and time that is acceptable to the technical staff and a TA. If a make-up is not permitted, the student will receive a grade of zero for the experiment.

Students will have the opportunity to discuss the experiments with the instructor during the lab period. This is an oral evaluation which will constitute the pre-lab component of the grade, and will be assessed based on students understanding of the chemistry and required techniques of the lab. As well students are encouraged to develop their ability to ask appropriate questions and discuss the chemistry.

**Formal Lab Report:** Two reports will be prepared as a formal, scientific document, with appropriate writing style, scientific language, and publication-quality figures and data interpretation. This is a key component of the course for its importance in developing the skills needed for effective written expression and the clear and concise communication of a piece of scientific work.

**Short Reports:** All other labs will include an informal short report, due on the Monday after the lab is complete, by 8:30 am. The templates for these will be provided through the course web-site.

**Poster Session:** This session will occur during the final week of the course, and be undertaken as pairs. Each group will choose a paper from the literature (either their own, or one from a list provided by the instructors). The students will read, summarize, and present the results of the paper, in relation to one of the labs they have completed.
Report Submission: All reports must be submitted in electronic form as PDF files using the corresponding drop boxes enabled in Avenue to Learn. As all submitted documents are subject to originality verification, it is necessary that you prepare your report using a word processing program, photographs of printed or handwritten pages will not be accepted.

The prelab for EVERY lab will consist of:

- Date and Title
- Hypothesis
- A list of reagents and balanced reactions.
- A FLOW CHART, indicating your plan of attack. It should be succinct. Any equations, and physical characterization techniques that apply.
- A Table in which you will record your data.
- A list of potential hazards and safety issues.

CHEM 2LB3 – Additional Course Notes for 2017

Course Changes. The instructor and university reserve the right to modify elements of the course during the term. The university may change the dates and deadlines for any or all courses in extreme circumstances. If either type of modification becomes necessary, reasonable notice and communication with the students will be given with explanation and the opportunity to comment on changes. It is the responsibility of the student to check their McMaster email and course websites weekly during the term and to note any changes.

Missed Academic Work. In the event of an absence for medical or other reasons during one of the midterm tests, students should review and follow the Academic Regulation in the Undergraduate Calendar “Requests for Relief for Missed Academic Term Work”. You may report your absence, using the McMaster Student Absence Form (MSAF, https://pinjap01.mcmaster.ca/msaf), without documentation. Absences for a longer duration or for other reasons must be reported to your Faculty/Program office, with documentation, and relief from term work may not necessarily be granted. When using the MSAF, report your absence to vargas@chemistry.mcmaster.ca. Within 2 days you will be informed whether relief was approved for the missed midterm.

Copyright. In this course you will have access to material that is subject to copyright laws. This includes (but is not limited to) the textbook and all resources developed by current and past instructors of the course. You are not allowed under any circumstance to share or redistribute these materials in any printed or electronic form without the explicit written consent of the copyright holder (the publisher, an instructor, etc.). This includes posting any course material on Internet bulletin boards, course repositories, social networks, etc.

Office Hours. There is no scheduled office hour for this course but students are always welcome to approach the instructor with questions or concerns. However, it is strongly encouraged that all questions regarding the course be initially posted in Avenue to Learn for the benefit of the whole class. Answers to questions will be posted within three working days. If you need to meet the instructor in person, try to book an appointment at least one day in advance through Avenue to Learn.

Instructor Availability. If the instructor is unable to attend a particular laboratory or classroom session, he may appoint a graduate teaching student to perform tasks such as the oral evaluations for the day.
List of experiments for 2LB3 - 2017 Synthetic-Inorganic Style (ABB 402)

1. **Catenation, Interhalogens and Positive Oxidation States in Group 17: [NMe₄][I₃], [NMe₄][ICl₂] and [NMe₄][ICl₄]**
   Area: Inorganic Chem: Poly and Interhalogens; Positive oxidation states and catenation in group 17 Raman spectroscopy
   Skills: Standard synthetic techniques (stirring and filtration) Reaction with Cl₂ gas
   Characterization by IR spectroscopy Characterization by Raman spectroscopy
   (Formal Report - due March 1, 2017)

2. **An electrochemical synthesis of [PPh₃H₂][Ga₂Br₆].**
   Area: Inorganic/Physical/Analytical Chem: Main group anions Redox chemistry
   Skills: Electrochemical synthesis (for Ga₂Br₆ dianion - under N₂ with Ga electrode made in lab from molten Ga)
   IR (and Raman provided) for [PPh₃H₂][Ga₂Br₆]
   (Short write-up)

3. **Isolation and electrochemistry of C₆₀.**
   Area: Organic/Inorganic/Physical/Analytical Molecules + Materials Chem: Allotropes of carbon, and solution electrochemistry
   Skills: Soxhlet extraction Column chromatography Distillation to remove a high boiling solvent (o-C₆H₄Cl₂)
   Cyclic voltammetry
   (Short write-ups – Due after Day 2, and Day 3)

4. **Synthesis of a “Weakly-Coordinating” Anion: Na[B{C₆H₃(CF₃)₂-3,5}₄]**
   Area: Organic/Inorganic Chem: Boranes and a borate anion, “non-coordinating anions”.
   Skills: Simple introduction to vacuum line techniques (1ˢᵗ vac line expt) Grignard reaction – unusual preparation using iPrMgBr.
   Interpretation of ¹H, ¹³F and (¹¹B) NMR spectra.
   (Short write-up – Due after Day 2)

5. **Tin and Lead Chemistry – SnCl₂ and [HPy]₂[PbCl₆].**
   Area: Inorganic Chem: Sn and Pb (MII and MIV)
   Inert pair effect and redox chemistry
   Skills: Slow addition (via dropping funnel) and reflux under N₂.
   Reaction at 0 °C under Cl₂.
   Quick tests of reducing and oxidising ability of Sn⁺Ⅱ and Pb⁺Ⅳ.
   (Short write-up)
6. **Silicon(IV) and Tin(IV) Compounds and Silly Putty**
   Area: Organic/Inorganic Molecules + Materials Chem: Inorganic Polymers
   Condensation of dialkyl-silanediol units is carried out to form silicone oil and the formation of “silly putty”. A 6-coordinate tin(IV) complex with acetylacetone (acac) is formed and characterized by $^1$H NMR and IR spectroscopy.
   Skills: General synthesis (MgSO$_4$, filtration, extraction, reflux, rotary evaporator, etc.) Characterization of cyclic products by IR and m.p.
   (Short write-up)

7. **Preparation of Ph$_2$P(CH)$_2$PPh$_2$ using Sodium in Liquid NH$_3$.**
   Area: Inorganic/Organic Chem: Electrides and alkalides Phosphorus chemistry
   Skills: Low temperature synthesis
   Synthesis under N$_2$
   Synthesis in anhydrous liquid NH$_3$. Extraction and recrystallization
   Characterization by $^1$H and $^{31}$P NMR (spectra for crude and recrystallized product will probably just be provided)
   (Short write-up)

8. **Creation of a Dye-sensitized TiO$_2$ Solar Cell + PEM-FCs**
   Area: Physical/Materials/Inorganic Chem: Extraction of dye from berry juice, complexation with TiO$_2$ film
   Skills: Assembly of electrodes (ITO + graphite-coated TiO$_2$/Berry Juice)
   Testing performance of Nafion-based cells using H$_2$ and methanol(aq)
   Using power supplies & multimeters
   (Short write-up – Due after Day 2)

9. **HPCL**
   Area: Analytical Chem: Detecting Caffeine
   Skills: Use of High Pressure Liquid Chromatography
   Data analysis and interpretation relative to standards
   (Short write-up – joint with GC)

10. **GC**
    Area: Analytical Chem: Detecting Caffeine
    Skills: Use of Gas Phase Chromatography
    Data analysis and interpretation relative to standards
    (Short write-up – joint with HPLC)

11. **Building your own Spectrophotometer**
    Area: Physical/Analytical Chem: Detecting Iron content using calibration curves
    Skills: Assembly of components; light source, filters, detector (hand-held GLX)
    Preparation of known Fe$^{3+}$(aq) solutions for calibration of instrument
    Analysis of “unknown” using student set-up.
    (Short write-up)
12. **Determining the Joule Thompson Coefficients for Carbon Dioxide**  
   Area: Physical/Materials Chem: Measuring the temperature change upon expansion of gas  
   Skills: use of digital pressure gauge and emf readings for temp. measurement  
   (Formal Report - due March 29th)

13. **GC-MS Investigation of Arson Case**  
   Area: Physical/Materials Chem: Detecting components in gasoline for identification of gas stations  
   Skills: “Finger-printing” using GS-MS traces  
   (Short write-up)

14. **Enzyme Kinetics**  
   Area: Physical/Materials Chem: Calculate the $k_{cat}$ and $v_{max}$ parameters for chymotrypsin from experimental data acquire at various temperatures.  
   Skills: UV-vis spectroscopy, steady-state approximation  
   (Short write-up)

15. **Layering of Gold Nanoparticles**  
   Area: Materials/Physical Chem: Preparation of Gold colloid by reduction of $\text{AuCl}_3$ Layering using crosslinking agents  
   Skills: UV-Vis, SEM  
   (Short write-up)

16. **Evaluating the Performance of a Stirling Engine**  
   Area: Physical/Materials Chem: measurement of work done by a simple heat engine as a function of temperature gradient  
   Skills: assembly of engine and tacometer, calculation of efficiency  
   (Short write-up)
Academic Dishonesty

Academic dishonesty is to knowingly act or fail to act in a way that results or could result in unearned academic credit or advantage.

Wherever in the Policy an offence is described as depending on “knowingly”, the offence is deemed to have been committed if the person ought reasonably to have known.

It is your responsibility to understand what constitutes academic dishonesty. For information on the various kinds of academic dishonesty please refer to the Academic Integrity Policy, specifically Appendix 3, located at

https://www.mcmaster.ca/policy/Students-AcademicStudies/AcademicIntegrity.pdf

The following illustrates only three forms of academic dishonesty:

1. Plagiarism, e.g. submitting academic work that has been, entirely or in part, copied from or written by another person without proper acknowledgement, or, for which previous credit has been obtained. Transcribing passages from other sources in assignments is an example.
2. Improper collaboration in academic work.
3. Copying or using unauthorized aids in tests and examinations.

All submitted work is subject to normal verification that standards of academic integrity have been upheld (e.g., Google search, Turn-It-In etc.).

Approved by McMaster University Senate: May 12, 2002
Revisions approved by McMaster University Senate: April 10, 2013

I hereby acknowledge that I have read McMaster’s policy on Academic dishonesty.

Name: ___________________________ Signature: ___________ Date: ___________

Please make a photocopy of this page, sign and submit before or during the Laboratory session of January 9, 2017.