CHEM 5321  Spectroscopic Methods in Organic Chemistry  Spring, 2020

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Office hours:  11:00-12:30 TTh; other meetings can be arranged by appointment.

Books:  No specific textbook is required for the class, as the available choices have shortcomings, the class notes you will get are reasonably detailed, and many practice problems will be provided. However, a couple of books are recommended as options to consider as possible reference materials. There will be no assignments from any of these, but students in the past who have commented about them have found them at least somewhat useful. The most commonly chosen option is a paperback called Structure Determination of Organic Compounds, Tables of Spectral Data that provides many examples of spectroscopic (especially NMR) data for various types of compounds. The most recent (4th) edition of this book was published in 2009 and was authored by Pretsch, Bühlmann, and Badertscher (ISBN 3540938095). An earlier edition would also be ok. These can be had at reasonably low cost—especially used/older copies. They receive good reviews for what they are, but they do not offer practice problems, explanations, or details of theory.

There are more traditional books. One is Spectrometric Identification of Organic Compounds, now in its 8th edition, by Silverstein, Webster, Kiemle, and Bryce (2014, ISBN-10: 0470616377). This edition is improved by inclusion of more practice problems, as well as updating of the content. Unfortunately, most of the first 120 pages are holdovers from old editions that focus extensively on EIMS and IR, and are of limited use nowadays. There is also nothing at all on UV or CD. On the other hand, the NMR portion is much improved, and there are many practice problems. There are 2-3 other options out there (e.g., Crews, et al. 2010), but each has its drawbacks. Such books tend to be more expensive, but used or international versions can be had. Any such book may serve as a good source of additional information, explanations, examples, illustrations, and problems.

Most of the necessary material will be provided via a set of course notes (see below), and nowadays there are a lot of relevant web resources, too. Links to such resources will be posted on the course ICON site. We will focus heavily on spectral interpretation, and this is best learned by covering basic principles and trends, discussing examples, and *working problems*. Because of this emphasis on applications, we will not cover theoretical aspects in the depth that they deserve. A “traditional” text like those above can provide detailed explanations of theory and instrumentation beyond the level of coverage in the course.

Course notes: Copies of the course notes (i.e., Powerpoint slides used in class) will be provided for download (in pdf format on ICON) as the semester progresses. These will be made available before they are covered in class, so that students may add written comments to them during lectures and, ideally, look them over before class. However, some changes, deletions, and additions tend to be made during the semester in an effort to keep the course updated, clarify points, and improve the quality of the notes, so the entire set is not made available at the beginning. Because of the value of spending as much of our class time as possible on problem-solving, not all of the notes will be covered in depth in class—some parts will skinned briefly, while others may simply be assigned as reading or reference material.

Class meetings:  9:30-10:45 AM TTh in room C139 PC. It may be necessary to cancel or reschedule one or two lectures during the semester due to conflicts. If so, advance notice will be given in class. Exams will be held outside of the regular class time, and this should give us some flexibility in making up any lost lecture periods. Because 75 minutes is a long time to sit and focus, we have traditionally taken a 5-minute break in the middle of the class and extended the period by 5 minutes. Unless someone has an objection to this practice, I plan to do the same this year. Logistics for this will be discussed in class and will depend on student schedules (and on whether there is a class immediately after ours in the same room).

Once we get to a suitable point in the notes, we will alternate between covering further notes and working through problems during class meetings. Past ACE data indicate that students view in-class problem-solving as a particularly valuable element of the course, so efforts will be made to dedicate as much of our class time to
this as possible. Attendance will not be recorded, but attendance and class participation will make the class more useful for everyone. Please do not be concerned about asking any question--all questions are welcome. Sometimes the simplest questions are the best...

**Exams:** There will be two mid-term exams and a final. Times, dates, and places will be arranged in class to accommodate student schedules. Exam I is typically held sometime during the week before Spring Break, while Exam II is late in the semester. *Exams will be open-book/open-notes and cumulative,* and will consist almost entirely of spectral interpretation and related types of problem-solving. A large block of time is needed for each exam, so they are usually held in the evenings.

**Course grades:** Grades will be based on exam performance and +/- grading will be used if appropriate based on the distribution of scores. The two mid-term exams will each be worth 30 percent of the final grade, while the final exam will be worth 40 percent. Partial credit will be given in the grading process. Grades will be curved and assigned depending on the performance of this year's class on the exams.

Problem sets and practice exams will be assigned to build experience with different kinds of situations and data sets, but will not be graded. These items and answer keys will be posted on ICON. Most, if not all, of these problems will be discussed in class. In fact, efforts will be made to work most of them in class before the *keys* are posted so that they can be used to maximum effect in developing problem-solving skills.

**Course content:** We will cover the most commonly used spectroscopic and spectrometric techniques in organic structure elucidation, with the exception of X-ray crystallography. The vast majority of time will be spent on NMR (ca. 75%) and MS methods (ca. 20%), their practical applications, and interpretation of data generated using these techniques. Exam I will focus on interpretation of standard $^1$H and $^{13}$C NMR data, while Exam II will *build upon knowledge from Exam I* by bringing in more sophisticated NMR techniques (including 2D NMR), and MS. The Final Exam will be comprehensive, and will include problems of the same types found on Exams I and II, along with coverage of a few additional minor topics.

I. NMR Spectroscopy
   A. General principles
   B. $^1$H NMR
   C. $^{13}$C NMR
   D. Other Nuclei
   E. FT NMR pulse sequences and relevant $"1D"$ NMR experiments
   F. 2D NMR

II. Mass Spectrometry (MS)
   A. Electron Impact MS
   B. Fragmentation
   C. High Resolution MS
   D. Soft-ionization techniques (ESI, CI, MALDI, etc.)
   E. MS-MS and other experiments

III. Selected Aspects of IR, UV, and CD Spectroscopy Relevant to Organic Chemistry

A few notes about the class. The main **objective** of this course is to develop students' skills in interpretation of spectroscopic data to facilitate their research progress. While we will not consider NMR or MS instruments or experimental protocols as "black boxes", the most critical topics for organic chemists are knowing what kind of data you need for your situation, how best to get them, and how to interpret them—this will be our focus.

Exams will be open book and open notes, but this will not help much if you do not *understand* the nuances of the data. Tables of NMR data, e.g., can be useful in giving you some ideas or supporting your thoughts about a proposed structure, but the answers will not lie there. Flipping through pages of data is only useful if you have already figured out something about what you are looking for.

Many of the principles in understanding NMR or MS interpretation come down to fundamentals that you learned in undergraduate organic chemistry. Concepts of electronegativity, electron density, polarity, resonance, bonding, valence, functional groups, H-bonding, isotopes, stereochemistry, conformation, bond angles, hybridization, aromaticity, solubility, $pK_a$, simple reaction tendencies, and others, are all important aspects that come into play in this course in one way or another. Naturally, we will add some specialized principles and terms, but a firm understanding of these principles will help a lot....
Absences and Attendance
Students are responsible for attending class and for contributing to the learning environment of a course. Students are also responsible for knowing their course absence policies, which will vary by instructor. All absence policies, however, must uphold the UI policy related to student illness, mandatory religious obligations, including Holy Day obligations, unavoidable circumstances, or University authorized activities (https://clas.uiowa.edu/students/handbook/attendance-absences). Students may use this absence form to aid communication; the instructor will decide if the absence is excused or unexcused (https://clas.uiowa.edu/sites/default/files/ABSENCE%20EXPLANATION%20FORM2019.pdf).

Academic Integrity
All undergraduates enrolled in courses offered by CLAS have, in essence, agreed to the College's Code of Academic Honesty. Misconduct is reported to the College, resulting in suspension or other sanctions, with sanctions communicated with the student through the UI email address (https://clas.uiowa.edu/students/handbook/academic-fraud-honor-code).

Accommodations for Disabilities
UI is committed to an educational experience that is accessible to all students. A student may request academic accommodations for a disability (such as mental health, attention, learning, vision, and physical or health-related condition) by registering with Student Disability Services (SDS). The student is then responsible for discussing specific accommodations with the instructor. More information is at https://sds.studentlife.uiowa.edu/.

Administrative Home of the Course
The College of Liberal Arts and Sciences (CLAS) is the administrative home of this course and governs its add/drop deadlines, the second-grade-only option, and related policies. Other colleges may have different policies. CLAS policies may be found here: https://clas.uiowa.edu/students/handbook.

Communication and the Required Use of UI Email
Students are responsible for official correspondences sent to the UI email address (uiowa.edu) and must use this address for all communication within UI (Operations Manual, III.15.2).

Complaints
Students with a complaint about a course should first visit with the instructor or course supervisor and then with the Chair of the department or program offering the course; students may next bring the issue to CLAS in 120 Schaeffer Hall. For more information, see https://clas.uiowa.edu/students/handbook/student-rights-responsibilities.

Final Examination Policies
The final exam schedule is announced around the fifth week of classes; students are responsible for knowing the date, time, and place of a final exam. Students should not make travel plans until knowing this information. No exams of any kind are allowed the week before finals. Visit https://registrar.uiowa.edu/final-examination-scheduling-policies.

Nondiscrimination in the Classroom
UI is committed to making the classroom a respectful and inclusive space for all people irrespective of their gender, sexual, racial, religious or other identities. Toward this goal, students are invited to optionally share their preferred names and pronouns with their instructors and classmates. The University of Iowa prohibits discrimination and harassment against individuals on the basis of race, class, gender, sexual orientation, national origin, and other identity categories set forth in the University’s Human Rights policy. For more information, contact the Office of Equal Opportunity and Diversity (diversity.uiowa.edu).

Sexual Harassment
Sexual harassment subverts the mission of the University and threatens the well-being of students, faculty, and staff. All members of the UI community must uphold the UI mission and contribute to a safe environment that enhances learning. Incidents of sexual harassment must be reported immediately. For assistance, please see https://osmrc.uiowa.edu/.