Chemistry 636 001 (2 credits) Spring 2020 [1204]

Topics in Chemical Instrumentation:
Introduction to NMR

Course Designations: Advanced level; physical science breadth; counts as L&S credit
Requisites*:* Graduate or professional student standing

Lecture:	Tuesdays, 8.50 a.m. to 9.40 a.m.
Location:	room 2311
Demo/lab sessions:	room 2224
Textbook:	"High-Resolution NMR Techniques in Organic Chemistry," by Timothy D.W. Claridge, Pergamon Press, 3rd edition, 2016. Free electronic versions of the textbook are available online from the library.
***Instructor*:**	Dr. Heike Hofstetter, Office: 2210B, 262-7536, hofstetter@chem.wisc.edu
Course webpage:	https://canvas.wisc.edu/courses/189837
***Office hours*:**	By appointment
Demo/lab sections:	Will be determined during first lecture period

Course Description and Goals:

This course will instruct students on the theory and practice of NMR spectroscopy. It is a full semester course, consisting of 15 hours of lecture and 30 hours of laboratory instruction. Additionally, homework sessions will be used to independently apply the material covered in lectures and demo sessions. Enrollment will be limited based on available instrumentation for lab exercises.
Practical aspects of NMR spectroscopy will be emphasized but some fundamental NMR theory will also be explored. The course provides in-lab training and laboratory experience. Students will be introduced to low- and high-field instrumentation in the facility, in both manual and automation mode. Upon completion of the course, students should be able to efficiently and independently use the facility instrumentation for a set of common NMR experiments. The fundamentals covered in this course will provide the necessary background for students intending to continue on to more advanced NMR techniques.

Learning outcomes:

Students who complete this course will be able to:

- Apply theoretical, conceptual, and observational knowledge of NMR methodology to the analysis and solution of chemical problems in graduate-level research.
- Compare and contrast experimental approaches pertinent to NMR analysis of original research.
- Demonstrate competence in collection and interpretation of NMR data.

- Communicate clearly and articulately knowledge, findings, and interpretations in oral presentations.

Course structure:

This 15-week class includes face-to-face and blended sessions. The credit standard for this course is met by an expectation of a total of 90 hours of student learning activity with the courses learning activities (45 hours per credit), which include regularly scheduled instructor/student meeting times (lecture and demo sessions), reading, problem sets, labs, and homework as described in the syllabus

TENTATIVE SCHEDULE

wk	Date	Lecture Outline	Lab	Spectrometer
1	1/21	Meeting during 1st week of class Syllabus and organization of class, Lab assignments	Installation of MNova	
2	1/28	Instrumentation and safety Principles of NMR: hardware, lab/rotating frames	Data workup: MNova	n/a
3	2/4	The NMR spectrum: Resolution and shimming → conjugate pairs: AQ ∝ 1/Δν, the NMR signal.	Introduction to ^{1}H 1D NMR	Eos
4	2/11	Recording a spectrum – 1D 1H-NMR.	^{1}H 1D NMR and resolution, filters and folding.	Eos
5	2/18	1D 13C NMR: sensitivity, quantitation, coupling	^{13}C 1D NMR	Eos
6	2/25	13C NMR: editing and signal enhancement	Advanced ^{13}C 1D NMR: polarization transfer (DEPT, INEPT, APT, DEPTQ)	Eos
7	3/3	Spin echoes and polarization transfer	Other nuclei: ^{19}F, ^{31}P, ^{119}Sn (+ ^{11}B, ^{29}Si.…)	Eos
8	3/10	Other nuclei	Automation: IconNMR	Artemis & Callisto (in automation)
9	3/17	SPRING BREAK		Eos
	3/24	Introduction to 2D NMR - HSQC	**Midterm lab exam**	
10	3/31	Homonuclear correlations: COSY, TOCSY	HSQC and assignments in MNova	Eos, Artemis, Callisto

11	4/7	More heteronuclear 2D: HMBC	Homonuclear 2D NMR: COSY and TOCSY	Eos, Artemis, Callisto
12	4/14	Processing in MNova, Relaxation and Selective 1D experiments (tocsy 1d)	Heteronuclear 2D NMR: HMBC (n-bond) Assignments in MNova Publishing NMR tables using MNova	Eos, Artemis, Callisto
13	4/21	Stereochemistry, ^{1}H-^{1}H distances (noesy, roesy)	T_1 relaxation and TOCSY1D	Eos
14	4/28	Project presentations	NOESY1D and ROESY1D	Eos, Artemis, Callisto
15	5/5	Finish up – project presentations	**Final lab exam**	Any/all spectrometers

Chemistry Instrument Center, MR Facility Personnel:

Director, MR Facility	Dr. Charlie Fry	Rm 2201a	262-3182
Assoc. Dir., MR Facility	Dr. Heike Hofstetter	Rm 2201b	262-7536
Teaching Assistant	Logan Vine	lvine@wisc.edu	

Grading:

Pre-lab quiz	10%
Attendance/work at demo session	10%
Practice sessions	5%
Homework	35%
Project presentation	10%
Mid-term lab exam	10%
Final lab exam	20%

There will be one 50 min lecture session each week.

In addition, you are expected to attend lab "demo" sessions where experiments for the respective homework assignments will be demonstrated on the NMR instrumentation and further discussed. Please make sure to answer the 2 pre-lab quiz questions on learn@UW prior to each lab demo. Those will count towards your practice sessions grade.

You will then have 1.5h of "practice" time per week to complete your homework assignments.

Homework assignments are due as specified on the handouts, generally one week after they are handed out, at the beginning of the lab period. All assignments will be submitted online. The total score for each homework will be 30 points, with a weighted penalty (5 points/day) for late homework. Carefully go over each homework assignment prior to sitting down at the spectrometer. Understand what the assignment requires you to do and develop a strategy *in advance* (!) to meet the goals of the assignment. This will significantly reduce homework time. Please submit homework with the spectra that are specifically asked

for first followed by answers to all short answer questions. After the questions you should include all other spectra taken.

During the 2nd part of the class experiments will be run on an "unknown" of your choice. A complete dataset, including assignments, will be acquired. Each student will present results of his/her work in a very short presentation (power point or handout) during the last (2) lecture periods. These presentations will be graded.

There will be two exams on the spectrometer, one mid-term and one final. These are open-book exams, but there is a time limit. No phones can be used during the exam.

Other resources:

The course website (under https://canvas.wisc.edu/courses/189837) will feature important announcements, links to homework assignments, pre-lab questions, and lecture slides for downloading. Check the site regularly, especially if you miss a class period and make sure to register to receive notifications about updates.

The main ***MR Facility Website*** (https://nmr.chem.wisc.edu) contains a wealth of information about instrumentation, software, experimental setup, etc. Be sure to check it out! This has use beyond this course and will guide you through many future NMR experiments.

Two computers in room 2219 and four computers in room 2224 are available for homework assignments. However, all assignments can be completed on your own personal device.

MestreNova Site Software and License Distribution:

From the MNova website (http://mestrelab.com/) download the software. The MR Facility uses v.14.01. Follow the instructions found on http://www.chem.wisc.edu/~cic/nmr/Guides/MNova/MNova-install.htm for installation of the program and the license file. The v.9 license file will work for newer versions.

NMR data access:

Data of the all spectrometers is located on castor.chem.wisc.edu in your user folder. You have to be connected to the chem vpn or Castor will not allow a connection. You can download your data using sFTP (WinSCP or FileZilla work well). Follow the instructions described here for Windows or Mac to connect to Castor https://www.chem.wisc.edu/~cic/nmr/Guides/Ba3vug/Castor_connect.pdf

Miscellaneous:

The key to the "Routine NMR lab" (room 2224) is also the building key, CBA1. Non-Chemistry students may obtain this key with a card (signed by facility personnel) from the building manager Jeff Nielsen.

Your chem logon and password will work on all spectrometers (if it does not please let the NMR staff know). Non-chemistry students will be assigned a logon and password.

The MRF facility charges $5 per hour for use of the 400 MHz spectrometer. The 500 MHz spectrometers are charged at $6 per hour. After you are done with the course, the time that you have used on all spectrometers for the course will be charged to your major advisor. Please see me if you don't have a major advisor or if you are taking the course purely to gain knowledge in the field of NMR.

Students with Disabilities:

McBurney Disability Resource Center syllabus statement: "The University of Wisconsin-Madison supports the right of all enrolled students to a full and equal educational opportunity. The Americans with Disabilities Act (ADA), Wisconsin State Statute (36.12), and UW-Madison policy (Faculty Document 1071) require that students with disabilities be reasonably accommodated in instruction and campus life. Reasonable accommodations for students with disabilities is a shared faculty and student responsibility. Students are expected to inform faculty [me] of their need for instructional accommodations by the end of the third week of the semester, or as soon as possible after a disability has been incurred or recognized. Faculty [I], will work either directly with the student [you] or in coordination with the McBurney Center to identify and provide reasonable instructional accommodations. Disability information, including instructional accommodations as part of a student's educational record, is confidential and protected under FERPA." http://mcburney.wisc.edu/facstaffother/faculty/syllabus.php

Academic Integrity and Misconduct:

By enrolling in this course, each student assumes the responsibilities of an active participant in UW-Madison's community of scholars in which everyone's academic work and behavior are held to the highest academic integrity standards. Academic misconduct compromises the integrity of the university. Cheating, fabrication, plagiarism, unauthorized collaboration, and helping others commit these acts are examples of academic misconduct, which can result in disciplinary action. This includes but is not limited to failure on the assignment/course, disciplinary probation, or suspension. Substantial or repeated cases of misconduct will be forwarded to the Office of Student Conduct & Community Standards for additional review. For more information, refer to https://conduct.students.wisc.edu/academic-integrity/

Diversity & Inclusion

Institutional statement on diversity: "Diversity is a source of strength, creativity, and innovation for UW-Madison. We value the contributions of each person and respect the profound ways their identity, culture, background, experience, status, abilities, and opinion enrich the university community. We commit ourselves to the pursuit of excellence in teaching, research, outreach, and diversity as inextricably linked goals.

The University of Wisconsin-Madison fulfills its public mission by creating a welcoming and inclusive community for people from every background – people who as students, faculty, and staff serve Wisconsin and the world." https://diversity.wisc.edu/