
Topics in Chemical Instrumentation:
Introduction to NMR

- Lecture:** Monday 11 a.m. to 11.50 a.m. room B357
- Textbook:** "High-Resolution NMR Techniques in Organic Chemistry," by Timothy D.W. Claridge, Pergamon Press, 2nd edition, 2008. 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://uwmad.courses.wisconsin.edu/d21/home/3361190> or log onto learn@UW → CHEM 636
- Office hours:** By appointment
- Lab sections:** Will be determined

Course Description and Goals: This course is the first of two NMR instrumentation classes currently offered by the Chemistry MR Facility. Practical aspects of NMR spectroscopy will be emphasized but some fundamental NMR theory will also be explored. The course provides in-lab training and practical 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 number 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.

TENTATIVE SCHEDULE

| wk | Date | Lecture Outline | Lab | Spectrometer |
|----|---------------------|--|---|-------------------------------------|
| 1 | 9/5 Labor Day | Meeting during 1 st week of class Syllabus and organization of class, Lab assignments | Installation of MNova | |
| 2 | 9/12 | Instrumentation and safety Principles of NMR: hardware, lab/rotating frames | Data workup: MNova | n/a |
| 3 | 9/19 | The NMR spectrum: Resolution and shimming → conjugate pairs: $AQ \propto 1/\Delta\nu$, the NMR signal. | Introduction to ¹ H 1D NMR | AC-300 (Athena) |
| 4 | 9/26 | Recording a spectrum – 1D 1H-NMR. | Filters and folding + ¹ H 1D NMR and resolution | Athena and Avance- 400 (Artemis) |
| 5 | 10/3 | 1D ¹³ C NMR: sensitivity, | ¹³ C 1D NMR | Avance-500P |

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| | | quantitation, coupling | | (Persephone) |
| 6 | 10/10 | ¹³ C NMR: editing and signal enhancement | Advanced ¹³ C 1D NMR: polarization transfer (DEPT, INEPT, APT, DEPTQ) | Avance-500C (Callisto) |
| 7 | 10/17 | Spin echoes and polarization transfer | Other nuclei: ¹⁹ F, ³¹ P, ¹¹⁹ Sn (+ ¹¹ B, ²⁹ Si....) | Persephone |
| 8 | 10/24 | Other nuclei | Automation: IconNMR and VNMRJ | Artemis & Hermes (in automation) |
| 9 | 10/31 | Introduction to 2D NMR - HSQC | Midterm lab exam | Persephone |
| | 11/7 | Homonuclear correlations: COSY, TOCSY | HSQC and assignments in MNova | Artemis |
| 10 | 11/14 | More heteronuclear 2D: HMBC | Homonuclear 2D NMR: COSY and TOCSY | Callisto |
| 11 | 11/21 | Processing in MNova, Relaxation and Selective 1D experiments (tocsy 1d) | Heteronuclear 2D NMR: HMBC (n-bond) Assignments in MNova Publishing NMR tables using MNova | Artemis |
| 12 | 11/28 | Stereochemistry, ¹ H- ¹ H distances (noesy, roesy) | T ₁ relaxation and TOCSY1D | Artemis/Persephone |
| 13 | 12/5 | Project presentations | NOESY1D and ROESY1D | Persephone |
| 14 | 12/12 | Finish up – project presentations | Final lab exam | Any/all spectrometers |

Chemistry Instrument Center, MR Facility Personnel:

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|--------------------------|----------------------|----------|----------|
| Director, MR Facility | Dr. Charlie Fry | Rm 2201a | 262-3182 |
| Assoc. Dir., MR Facility | Dr. Heike Hofstetter | Rm 2201b | 262-7536 |
| NMR Instr. Technologist | Dr. Lingchao Zhu | Rm 2237a | 262-8196 |
| Teaching Assistant | Manar Alherech | Rm | |

Grading:

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|---------------------------------|-----|
| Pre-lab work | 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 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.

During the 2nd part of the class experiments will be run on an “unknown”. A complete dataset, including assignments, will be acquired. Each student will present results of his/her work in a 2 min 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://uwmad.courses.wisc.edu/d2l/home/2961822>) 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://www.chem.wisc.edu/~cic/nmr/main.html>) contains a wealth of information about instrumentation, software, experimental setup, etc. Be sure to check it out!

One computer in room 2219 and five computers in room 2224 are available for homework assignments.

MestreNova Site Software and License Distribution:

From the MNova website (<http://mestrelab.com/>) download the software. The MR Facility uses v.10.0. 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 still work for v.10.

NMR data access:

Data for the all spectrometers (castor.chem.wisc.edu) in your user folder. Data from Athena will not be saved in your use folder but in a general “00_Athena” folder on castor. You have to be connected to the chem vpn or Castor will not allow a connection. You can either map castor as a network drive or download your data using sFTP. 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 CGF) from the building manager Jeff Nielsen.

Your chem logon and password will work on all spectrometers. Non-chemistry students will be assigned a logon and password.

The MRF facility charges \$3.22 per hour for use of the 400 MHz spectrometer. The 500 MHz spectrometers are charged at \$6.00 per hour. After you are done with the course, the time that you have used on Hermes and Artemis 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:

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. If you are a student with physical, learning, emotional, or psychological disabilities, you are encouraged to make an appointment with McBurney Disability Resource Center for assistance (<http://www.mcburney.wisc.edu>).

Students are expected to inform 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. I will work either directly with the student 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.

Academic Integrity and Misconduct:

Academic honesty is essential to the existence and integrity of an institution of higher education. The responsibility for maintaining that integrity is shared by all members of the academic community. Thus, academic misconduct will not be tolerated at this University. Any student caught cheating or attempting to cheat will be punished by automatically receiving a zero for that assignment. Academic misconduct includes copying from another's assignment, plagiarizing published materials and/or fabricating results. A second infraction will result in a failure for the course. Refer to the UW academic polices for more information on what constitutes academic misconduct (<http://www.students.wisc.edu/doso/students/>).