Chem. 753 Analytical Spectroscopy: Practical NMR

Spring 2023

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COURSE INFORMATION

Class Days: M/W
Class Times: 7:00-8:15 PM
Class Location: LH-249 (NMR Facility & CSL-222)
Office Hours: 6:00-7:00 PM (M/W)

Office Hours Location: GMCS-213C
Email Dr. David Onofrei to get NMR training:
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Course Overview

Chem. 753 Analytical Spectroscopy: Practical NMR is a graduate level course in NMR Spectroscopy. This class is focused on learning the practical aspects of NMR spectroscopy including the basics of NMR hardware, pulse sequence and experimental set-up, data processing and data interpretation. Although some theory will be taught, the primary focus of this class is on 'hands-on' training in NMR spectroscopy. The students will spend a considerable fraction of time collecting, processing and analyzing real NMR data with NMR software and some fraction of the class will be spent doing NMR training in the NMR lab. Data collection, data processing and data analysis will be covered. Magnetic Resonance has a plethora of different types of experiments; probe designs, pulse sequences, data collection techniques, and data simulation and modeling methods. The large variety of "methods" in magnetic resonance is often overwhelming for new scientists trying to learn how to setup and perform a specific experiment or process and analyze a specific set of data or spectra. The student's grade will come from two class NMR projects and presentations on a compound of choice. The primary goal of the class is to train the student to collect, process and interpret 1D and 2D NMR spectra and relaxation data and ultimately enable them to utilize NMR in their thesis research and future scientific research careers.

Student Learning Outcomes (SLO):

1) Basics of NMR, NMR Lab Safety, hardware overview, sample prep, student copy of NMR processing software.

2) 1D 1H NMR. Setup, ^{2}H lock and shim, processing (FT, EM, phase, zero filling, referencing, peak picking, baseline correction, integration, plotting electronic figure).

3) Pulse sequences, pulse programming and experimental design.

4) Advanced 1D 1H NMR**,** J-splitting, chemical shift, T_1 and T_2 measurement, FID analysis, Nutations, Spectral editing pulse sequences.

5) Heteronuclear NMR, ^{13}C, ^{31}P, ^{19}F, etc.

6) Advanced 1D NMR, solvent suppression, selective excitation, DEPT, APT.

7) Common 2D NMR, COSY, NOESY, HSQC, HMBC, INADEQUATE, etc.

8) Tricks for data collection and processing of 2D NMR, 2D-FT, window functions, apodization, linear prediction.

9) NMR self-diffusion measurements and DOSY methods.

10) Basic solid-state NMR techniques.

Enrollment Information

Prerequisites: There are no specific pre-requisites for this class, but it is a graduate level class, and some knowledge of General, Physical and Analytical chemistry is expected.

Course Materials

Book: *No required book, but there are a number of recommended optional materials listed below.*

Lecture Notes: Lecture notes are available upon request. These notes cover the major topic I will go over, **but may not include problems and examples done on the board, NMR demonstrations conducted with NMR software or 'hands on' training conducted in the NMR lab.** Schemes and schematics described on the board generally will not be posted. You will miss this material if you do not attend class. We will also cover some classic NMR journal articles that will be provided to the students prior to lecture on the topic. Please read these articles prior to class.

Recommended (Optional) Materials: Some Classic NMR Texts.

Principles of Nuclear Magnetism, by A. Abragam, Oxford, 1961; ISBN: 978-0-19-852014-6.

Spin Dynamics, by M.H. Levitt, Wiley, 2001; ISBN: 0-471-48922-0.

200 and More NMR Experiments, by S. Berger and S. Braun, Wiley, 2004; ISBN: 3-527-31067-3.

Experimental Pulse NMR: A Nuts and Bolts Approach, by E. Fukushima and S.B.W. Roeder, Perseus, 1981; ISBN: 0-201-62726-4.

Principles of Nuclear Magnetic Resonance in One and Two Dimensions, by R.R. Ernst, G. Bodenhausen and A. Wokaun, Oxford, 1987; ISBN: 0-19-855647-0.

Protein NMR Spectroscopy, by J. Cavanagh, W.J. Fairbrother, A.G. Palmer, M. Rance, N.J. Skelton, Elsevier, 2007; ISBN13: 978-0-12-164491-8.

Spectrometric Identification of Organic Compounds, by R.M. Silverstein, F.X. Webster and D.J. Kiemle, Wiley, 2005; ISBN: 0-471-39362-2.

Multidimensional Solid-state NMR and Polymers, by K. Schmidt-Rohr and H.W. Spiess, Academic Press, 1994; ISBN: 0-12-626630-1.

Course Structure and Conduct

This course will be taught in a combination of formats including lecture 'chalk talk' format, power point presentation, NMR software in class and some hands-on training at the NMR spectrometer and data collection by the student for projects. Lecture notes and presentations are available upon request. I expect this class to be somewhat informal where questions can be asked during class with discussion, during my office hours or following the lecture. The best advice for this class is to be proactive. Start Class Projects early and/or schedule a time to meet with me if you feel you are falling behind and take advantage of the optional and assigned reading.

Course Assessment and Grading

Projects: Due March 10 and May 5. Data for projects will need to be collected by the student. There will be 2 class projects that will involve collecting, processing and analyzing real NMR data and reporting the results as a paper with figures in a journal-like report. There will be two 15-minute presentations on the results that you have presented in your report. The final grade will be based on a maximum of **500 points**, distributed as follows: 2 reports (150 points each) and 2 presentations (100 pts each).
Letter Grade Assignment: The class is not curved. Letter grades will be assigned as follows: A (450 pts), B (400 pts), C (300 pts). If you cannot meet the project deadline you will be docked points so, please start your projects early.

Students with Disabilities

If you are a student with a disability and believe you will need accommodations for this class, it is your responsibility to contact Student Disability Services at (619) 594-6473. To avoid any delay in the receipt of your accommodations, you should contact Student Disability Services as soon as possible. Please note that accommodations are not retroactive, and that accommodations based upon disability cannot be provided until you have presented your instructor with an accommodation letter from Student Disability Services. Your cooperation is appreciated

Extra help and tips for Success

Help is available in a variety of forms.

- Work with your classmates on difficult material.
- Utilize office hours.
- Take advantage of the optional reading. The instructor can direct the student to a specific book chapter to help with a certain concept.

10 Musts to get a good grade:

- Attend all lectures.
- Read assigned and optional material, prior knowledge will help you become engaged in lecture and better comprehend material.
- Write questions down, and attend office hours.
- Take class projects seriously. Approach it as you would your thesis research.
- Discuss concepts with classmates, or study partner. Practice your presentation in front of your classmates prior to presentation day.
- Do not fall behind.
- Try to see the big picture. NMR is extremely complex. You will not grasp all of it.
- Be curious. Always ask why? Questions are welcome.
- Focus on understanding concepts, not memorization.
- Start early on class projects and presentation. It takes considerable time and effort to write a good report and/or give a good presentation.
- Remember the end goal is to learn practical NMR and how you can apply it to your research interests.