University of Wisconsin-Madison
CHEMISTRY 605, 3 credits
Spectrochemical Measurements
Spring 2020
General Course Information
MWF, Room 1315 Chemistry
12:05 PM-12:55 PM

Instructor:	Professor Jennifer M. Schomaker 8108 Shain Research Tower, Chemistry schomakerj@chem.wisc.edu
Office Hours:	Fridays from 1-2 PM; 8108 Shain
Websites:	Website: https://canvas.wisc.edu/courses/174952
Required Course info:	Instructional mode: face-to-face Credit hours are met by the Traditional Carnegie Definition

---

I. OFFICIAL COURSE DESCRIPTION

Mass spectrometry and applied nuclear magnetic resonance. Three lecture sessions per week. Prerequisites are either CHEM 562 or the consent of the instructor.

II. COURSE MATERIALS

Recommended Textbooks:

"Basic One- and Two-Dimensional NMR Spectroscopy," H. Friebolin, 5th Ed., VCH, 2010 (ISBN 3527327827).

"Tables of Spectral Data for Structure Determination of Organic Compounds," E. Pretsch, P Bülmann, M. Badertscher 4rd Ed. Springer Verlag, 2009 (ISBN 3540938095).

"Organic Structural Spectroscopy," J.B. Lambert, H.F. Shurvell, D.A. Lightner, R.G. Cooks, Prentice Hall, 1998.

"NMR Spectroscopy," H. Günther, 2nd Ed., John Wiley & Sons, 2005.

Course websites: The course will make extensive use of Canvas. Lecture notes, handouts, problem sets, reading assignments, and announcements will be posted to the course website regularly. You will also use Canvas to view your grades (https://canvas.wisc.edu/courses/174952).

Other websites and web sources that might be useful for extra problems and explanation are listed below; please feel free to let me know if you run across any others! If you are not comfortable with solving the types of NMR problems that you would encounter in a typical 2nd-semester undergraduate organic chemistry course, you should consider using some of these websites for additional practice.

http://www.chem.ucla.edu/~webspectra/
http://www3.nd.edu/~smithgrp/structure/workbook.html

http://www.chem.uci.edu/~jsnowick/organicspectroscopy/
Basic 2D NMR practice problems: http://pubs.acs.org/doi/pdf/10.1021/acs.jchemed.6b00007

III. LECTURE AND DISCUSSION

Reading: I've included background reading assignments in the accompanying list of topics that will be covered in the course. My primary goal is to give you the tools and strategies you require to elucidate structure and understand reaction behavior, not to teach you the theory or physical basis for the spectroscopic techniques discussed in class. If you are interested in obtaining a more in-depth understanding of the topics discussed in the course, I would recommend the reading assignments or taking Charlie Fry's Chem 637 course, if it is still being offered this summer.

Grading:

Problem sets (10 x 20 points each)	**200 points**
Exam 1	**150 points**
Exam 2 (final)	**150 points**
Class 'unknown' project	**100 points**
Class participation (quizzes, problem-solving sessions)	**100 points**

Problem Sets. There will be 10 problem sets during the course of the semester. These can be worked on in groups, but please be careful not to simply copy answers. Mastering spectroscopy requires a great deal of practice, so it is your best interest to attempt problems first on your own. Problem sets will be made available on Canvas at least a week in advance and should be turned in on the dates listed on the Tentative Agenda. I will look through the problem sets to ensure that you have made sufficient progress, assign a preliminary grade and hand the problem sets back on Friday. The class as a whole will then solve the problems- these sessions are an opportunity to rack up participation points. At the end of the class on Friday, you have the option of turning in your 'corrected' homework to get back 50% of the points you may have missed. You cannot get back these extra points if you opt to skip the Friday problem-solving session.

Quizzes. I will regularly pass out short 'quizzes' a lecture ahead of time. These are already posted on the Canvas website, but I will hand out paper copies in class, as we sometimes end up getting a bit off schedule. At the beginning of the next class period, I will ask for volunteers to solve the problems. This is another opportunity to earn participation points.

Class 'unknown' project. Sometime in late February, I will start assigning your unknowns. Heike has graciously agreed to collect a variety of conventional 1D and 2D NMR spectra for you, so you do not need to have taken Chem 636 or know how to operate the instruments (although we can provide you with FIDs if you wish to reprocess the data). Other data, such as a molecular weight, IR stretches or advanced 2D NMR data will be provided as needed.

You and a partner will work together to identify your unknown using both spectra and computational/modeling tools that may help you to support any tentative assignments. You are responsible for describing both the structure and the relative stereochemistry of your molecule. One week before your scheduled oral presentation to the class, you will turn in a written report describing how you analyzed your data and how you came to assign your proposed structure. This report will be made available to the class so that they have sufficient time to follow your logic prior to a final presentation you will make during the last few weeks of the course. You and your partner can split the duties of the final presentation any way you wish, but I suggest one person address functional groups present in the molecule and the 1D NMR data, while the second person discuss any 2D NMR and computational studies. More details will be forthcoming as the semester progresses.

Examinations: There will be only two exams this semester. One will be a 'mid-term', while the other can be given either during our scheduled Final Exam time or moved to a Saturday with agreement from the entire class. The first exam will be a take-home exam. For the second exam, you may bring any written materials you find useful to the exam. Calculators are fine, but computer devices and internet use are not allowed.

IV. LEARNING OUTCOMES

Course Learning Outcomes are included as a requirement for an upcoming audit of the university. Undergraduate and graduate learning outcomes are identical.

At the end of this course, students should be able to:

- Analyze and interpret a variety of spectroscopic data to deduce the chemical structure of an unknown compound.
- Choose the appropriate experiment(s) in a research setting to determine the structure and/or dynamic behavior of an unknown compound.
- Utilize modern techniques, skills and tools necessary for the practice.
- Examine technical literature, resolve ambiguities and develop conclusions.
- Communicate complex scientific ideas in a clear and understandable manner through written reports, oral presentations and discussion.
- Guide, mentor and support peers to achieve excellence in practice of the discipline.
- Work in multi-disciplinary teams and provide leadership on problems that arise.
- Foster safe, ethical, and professional conduct.

Tentative Agenda (Subject to change depending on the pace at which topics are covered)

Week	Date	Reading Material	Reich Handouts	Selected Topics
1	W Jan 22	**Pavia** Chapters 1-2 Lambert, Chapter 1 Lambert Ch. 8 (focus on IR)	Notes-02-IR-v23 2014	Introduction and course outline Comparison of important spectroscopic methods in use Electromagnetic spectrum Vibrational spectroscopy (IR, REACT-IR) IR group frequencies Structural analysis using IR
	F Jan 24	**Pavia** Chapter 3.1-3.10 Lambert Ch. 2, 3 Friebolin Ch. 1, 2.2, 6.2	05-HMR-v22-all 2014 5-HMR-1 5-HMR-2	NMR Introduction and experimental methods Simplified description of the NMR experiment ^{1}H integration and chemical shifts Symmetry considerations
2	M Jan 27	**Pavia** Chapter 3.11-3.12 Lambert Ch. 3 Friebolin Ch. 2.2	5-HMR-2, 9.5	**Quiz 1 (IR Practice)** Factors influencing chemical shift Curphy-Morrison tables and exceptions Spin-spin splitting, why it occurs, Pascal's triangle
	W Jan 29	**Pavia** Chapter 3.13-3.19 Lambert Ch. 4 Friebolin Ch. 3.1-3.3	5-HMR-3 5-HMR-3.3-3.10	First-order multiplets, size of couplings Two different couplings to one proton Substitution patterns in aromatics Leaning effects Determining whether a multiplet exhibits a first-order pattern **Problem Set 1 is due**
	F Jan 31	**Solve Problem Set 1**		**Solve Problem Set 1**
3	M Feb 3	**Pavia** Chapter 5 Lambert Ch. 4 Friebolin Ch. 3.1-3.3, 4	5-HMR-3.3-3.10	**Quiz 2** "Nutty" compounds More practice with 1st-order multiplets Multiplets that are not 1st-order
	W Feb 5	**Guest Lecturer** **Dr. Ilia Guzei**	Dr. Guzei Handout	X-ray Crystallography **Problem Set 2 is due**
	F Feb 7	**Guest lecturer**	**Solve Problem Set 2**	**Solve Problem Set 2**
4	M Feb 10	**Pavia** Chapter 4 Lambert Ch. 3.3-3.4 Friebolin Ch. 2.3, 3.4, 6.3	6-CMR-1.1 6-CMR-3.1-3.9 6-CMR-3.11, 4.2	**Quiz 3** ^{13}C NMR chemical shifts β,γ effects on chemical shift Ring size effects on chemical shift Effect of conjugation, charge effects, hydrogen bonding on ^{13}C chemical shifts, parameter tables
	W Feb 12	**Pavia** Chapter 8 Lambert Ch. 13-15 **Guest Lecturer**	Reich Handout 3	Introduction to Mass Spectrometry Presentation of data and nomenclature Isotopes, molecular formulas, High and low resolution techniques

		Dr. Martha Vestling		Different ionization techniques Analysis of ions, Different types of detectors Analyzing fragmentation patterns
	F Feb 14	**Guest Lecturer** **Dr. Martha Vestling**		Modern mass spec techniques, UW-Chemistry Mass Spec Center **Problem Set 3 is due**
5	M Feb 17	**Solve Problem Set 3** **Pavia** Chapter 4 Lambert Ch. 3.3-3.4 Friebolin Ch. 2.3, 3.4, 6.3	6-CMR-1.1 6-CMR-3.1-3.9 6-CMR-3.11, 4.2	**Solve Problem Set 3** Continue discussion of ^{13}C NMR ^{1}H-^{13}C couplings DEPT
	W Feb 19	**Pavia** Chapter 5 Lambert Ch. 4 Friebolin Ch. 4	5-HMR-3.8-3.10 5-NMR-3.13	**Quiz 4** Pople nomenclature for coupled spin systems Symmetry and magnetic equivalence Second-order effects in coupled systems: AX and AB patterns
	F Feb 21	**Pavia** Chapter 5	5-HMR-3.8-3.10 5-NMR-3.13 5-HMR-7.1-7.2 5-HMR-8.1-8.3	**Quiz 5** Second-order effects in coupled systems AX and AB Spectra
6	M Feb 24	**Guest Lecturer** **Dr. Desiree Bates**	Bates Handout	Computational methods for predicting chemical shift and coupling constants
	W Feb 26	**Pavia** Chapter 5	5-HMR-9.1-9.3 5-NMR-10.1-10.6 5-HMR-7.1-7.2 5-HMR-8.3	**Quiz 6** Solving AX_2 and AB_2 patterns ABX patterns Solving ABX patterns **Problem Set 4 is due**
	F Feb 28	**Solve Problem Set 4**		**Solve Problem Set 4**
7	M Mar 2	**Pavia** Chapter 5	5-HMR-11.1-11.2 5-HMR-12, 5-12.4 5-HMR-12.6-12.9, 12.15-12.16	**Quiz 7** $ABX_mY_nZ_o$ patterns ABX_3 patterns
	W Mar 4		5-HMR-12.19-20 5-HMR-13.1-13.5	**Quiz 8** $ABMX_3$ patterns, virtual coupling A_2X_2, AA'XX', AA'BB' patterns
	F Mar 6		5-HMR-13.1-13.6 5-HMR-16.1-16.8 5-HMR-14.1-14.3 5-HMR-15.1-15.5	**Quiz 9** Review and more detail on the factors that influence the size of couplings (2 bond coupling) Vicinal proton-proton coupling, three-bond coupling Determining conformations of rings using *J* values

			5-NMR-5.3-6.6 5-HMR-15.2-15.8 5-HMR-15.10 5-HMR-4.1-4.5	Long-range coupling, stereochemical determination Anistropic effects, Allylic coupling, Long-range coupling **Problem Set 5 is due**
8	M Mar 9 W Mar 11 F Mar 13	**Pavia** Chapter 10.1-10.3, 10.9-10.10 **Pavia** Chapter 10.1-3, 10.6-7 Schomaker basic 2D NMR **In-class portion of exam**	8-NMR (8-Tech-1.1-2.17) 8-NMR-9.1.1-9.3.3 8-NMR-9.1-9.6 **In-class portion of exam**	**Solve Problem Set 5** Finish coupling constant discussion Relaxation processes T_1 and T_2 in ^{1}H, ^{13}C, and other nuclei The Nuclear Overhauser Effect **Quiz 10** 2D Multinuclear NMR (quick discussion) **In-class portion of exam**
9	M Mar 23 W Mar 25 F Mar 27	**Jess Roberts** **Pavia** Chapter 10.8 **Pavia** Chapter 10.1-3, 10.6-7 Schomaker basic 2D NMR	**Solve Exam** 8-NMR-9.1-9.6 7-MULTI-1.1-4.8 7-MULTI-1.1-4.8 8-NMR-9.1.1-9.3.3 8-NMR-9.1-9.6	**Solve exam and turn in Exam 1 take-home portion** **Quiz 11** Isotopic labeling, isotope shifts Other useful nuclei: ^{19}F, ^{31}P, ^{10}B, ^{15}N, metals **Quiz 12** Continue multinuclear NMR The spin 1/2 nuclei, Quadrupolar nuclei, quadrupolar relaxation Other useful nuclei: ^{19}F, ^{31}P, ^{10}B, ^{15}N, metals **Problem set 6 is due**
10	M Mar 30 W Apr 1 F Apr 3	**Solve Problem Set 6** Schomaker-MULTINUC Schomaker-DYNAMIC	**Solve Problem Set 6** 11-MultiNuclear 8-TECH-3 to 8-TECH 6	**Solve Problem Set 6** **Quiz 13** Finish discussion of multinuclear NMR Quadrapolar Nuclei **Quiz 14** Dynamic NMR: Measurement of conformational and chemical exchange rates, Line broadening - variable temperature NMR spectra The Forsen experiment - saturation transfer **Problem set 7 is due**
11	M Apr 6 W Apr 8 F Apr 10	**Solve Problem Set 7** Schomaker-NMR Kinetics SCHOMAKER Notes	**Solve Problem Set 7** 8-TECH-3 to 8-TECH 6	**Solve Problem Set 7** Practical aspects of NMR kinetics, calculating thermodynamic parameters using NMR kinetics experiments, EXSY **Quiz 15** Finish NMR kinetics, ^{13}C Kinetic Isotope Effect Measurements **Written Report on Unknowns is due**

12	M Apr 13 W Apr 15 F Apr 17	SCHOMAKER Notes **Solve Problem Set 8** SCHOMAKER Notes	 **Solve Problem Set 8**	**Quiz 16/17** Other useful NMR techniques (DOSY, trNOE, STD) **Problem set 8 is due** **Solve Problem Set 8** Methods to determine absolute stereochemistry by NMR, X-ray, and ECCD
13	M Apr 20 W Apr 22 F Apr 24	Class Presentations Class Presentations Class Presentations	----------- ----------- -----------	Class Presentations Class Presentations Class Presentations
14	M Apr 27 W Apr 29 F May 1	Class Presentations Class Presentations Class Presentations	----------- ----------- -----------	Class Presentations Class Presentations Class Presentations
				Final unknown report is due the day of the final exam. **The Final Exam will be given at the regularly scheduled time.**