CHEMISTRY 605
Structure Determination Using Spectroscopic Methods
Spring 2017
General Course Information
MWF, Room 1315 Chemistry
12:05 PM-12:55 PM

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Websites: Learn@UW
http://www.chem.wisc.edu/areas/reich/chem605/

I. 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 Learn@UW. Lecture notes, handouts, problem sets, reading assignments, and announcements will be posted to the course website regularly. You will also use Learn@UW to view your grades. Professor Reich's website is still available, but most of the information on his website is also available on my Learn@UW page:

http://www.chem.wisc.edu/areas/reich/chem605/

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.ucalgary.ca/courses/351/Carey5th/Ch13/isp/index.html
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

II. 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 offered in the 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 Learn@UW 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. 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: I've decided to give only two exams this semester. One will be a 'mid-term', while the other can be given either during our schedule Final Exam time or moved to a Saturday with agreement from the entire class. These exams are "open book" - you may bring any written materials you find useful to the exam. Calculators are fine, but computer devices and internet use are not allowed. The suggested date and time for the first exam is **Saturday, March 11 at 10:00 AM**. You will have 6 hours to complete the exam, although hopefully, you should not require this much time! Please let me know as soon as possible if you have a conflict with this date.

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 18	**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 20	**Pavia** Chapter 3.1-3.10 Lambert Ch. 2, 3 Friebolin Ch. 1, 2.2, 6.2	Notes-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 **Pass out Problem Set 1: IR and basic NMR**
2	M Jan 23	**Pavia** Chapter 3.11-3.12 Lambert Ch. 3 Friebolin Ch. 2.2	5-HMR-2, 9.5	Factors influencing chemical shift Curphy-Morrison tables and exceptions Spin-spin splitting, why it occurs, Pascal's triangle
	W Jan 25	**Pavia** Chapter 3.13-3.19 Lambert Ch. 4 Friebolin Ch. 3.1-3.3	5-HMR-3 5-HMR-3.3-3.10	Multiplet quiz 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 due, pass out Problem Set 2**
	F Jan 27	**Solve Problem Set 1**		**Solve Problem Set 1**
3	M Jan 30	**Pavia** Chapter 5 Lambert Ch. 4 Friebolin Ch. 3.1-3.3, 4	5-HMR-3.3-3.10	"Nutty" compounds More practice with 1st-order multiplets Multiplets that are not 1st-order
	W Feb 1	**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	^{13}C NMR chemical shifts β,γ gamma effects on chemical shift ring size effects on chemical shift effect of conjugation, charge effects, hydrogen bonding on ^{13}C chemical shifts, parameter tables **Problem Set 2 due, pass out Problem Set 3**
	F Feb 3	**Solve Problem Set 2**		**Solve Problem Set 2**
4	M Feb 6	**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 quiz continue discussion of ^{13}C NMR

	W Feb 8 F Feb 10	**Pavia** Chapter 5 Lambert Ch. 4 Friebolin Ch. 4 **Solve Problem Set 3**	5-HMR-3.8-3.10 5-NMR-3.13	Multiplet quiz Pople nomenclature for coupled spin systems Symmetry and magnetic equivalence Second-order effects in coupled systems: AX and AB patterns **Problem Set 3 due, pass out Problem Set 4** **Solve Problem Set 3**
5	M Feb 13 W Feb 15 F Feb 17	**Pavia** Chapter 5 **Pavia** Chapter 5 **Solve Problem set 4**	5-HMR-3.8-3.10 5-NMR-3.13 5-HMR-7.1-7.2 5-HMR-8.1-8.3 5-HMR-9.1-9.3 5-NMR-10.1-10.6 5-HMR-7.1-7.2 5-HMR-8.3	Multiplet quiz Second-order effects in coupled systems AX and AB Spectra Solving AX_2 and AB_2 patterns ABX patterns Solving ABX patterns **Problem Set 4 due, pass out Problem Set 5** **Solve Problem set 4**
6	M Feb 20 W Feb 22 F Feb 24	**Pavia** Chapter 5 **Solve Problem Set 5**	5-HMR-11.1-11.2 5-HMR-12, 5-12.4 5-HMR-12.6-12.9, 12.15-12.16 5-HMR-12.19-20 5-HMR-13.1-13.5	Solve multiplet quiz $ABX_mY_nZ_o$ patterns ABX_3 patterns Solve multiplet quiz $ABMX_3$ patterns, virtual coupling A_2X_2, AA'XX', AA'BB' patterns **Problem Set 5 due, pass out Problem Set 6** **Solve Problem Set 5**
7	M Feb 27 W Mar 1 F Mar 3	 **Solve Problem Set 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 5-NMR-5.3-6.6 5-HMR-15.2-15.8 5-HMR-15.10 5-HMR-4.1-4.5	Solve multiplet quiz 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 Long-range coupling, stereochemical determination Anistropic effects, Allylic coupling, Long-range coupling **Problem Set 6 due, Pass out Problem Set 7** **Solve Problem Set 6**
8	M Mar 6	**Pavia** Chapter 10.1-10.3, 10.9-10.10	8-NMR (8-Tech-1.1-2.17)	Relaxation processes T_1 and T_2 in ^1H, ^{13}C, and other nuclei The Nuclear Overhauser Effect (NOESY, ROESY experiments) COSY, dqfCOSY, TOCSY

	W Mar 8 F Mar 10 S Mar 11	**Pavia** Chapter 10.1-3, 10.6-7 Schomaker basic 2D NMR **Solve Problem Set 7** **EXAM 1**	8-NMR-9.1.1-9.3.3	Continue discussion of 2D NMR techniques **Solve Problem Set 7** **EXAM 1**
9	M Mar 13 W Mar 15 F Mar 17	**Pavia** Chapter 10.4 Schomaker DEPT Schomaker basic 2D NMR **Solve Exam 1**	8-NMR-9.1-9.6 8-NMR-9.1-9.6 7-MULTI-1.1-4.8	^{1}H-^{13}C couplings DEPT Start of multinuclear NMR The spin 1/2 nuclei, Quadrupolar nuclei, quadrupolar relaxation **Pass out Problem Set 8** **Solve Exam 1**
10	M Mar 27 W Mar 29 F Mar 31	**Pavia** Chapter 10.8 Schomaker-MULTINUC **Solve Problem Set 8**	7-MULTI-1.1-4.8 11-MultiNuclear ---------	Isotopic labeling, isotope shifts Heteronuclear correlation (HMQC, HMBC) Other useful nuclei: ^{19}F, ^{31}P, ^{10}B, ^{15}N, metals Quadrapolar nuclei **Problem Set 8 due, pass out Problem Set 9** **Solve Problem Set 8**
11	M Apr 3 W Apr 5 F Apr 7	Schomaker-DYNAMIC Schomaker-NMRKinetics Schomaker-DYNAMIC Schomaker-NMRKinetics **Solve Problem Set 9**	8-TECH-3 to 8-TECH 6	Dynamic NMR: Measurement of conformational and chemical exchange rates, Line broadening - variable temperature NMR spectra The Forsen experiment - saturation transfer Case studies of understanding dynamic processes in solution Measuring rates of reaction using NMR kinetics **Problem Set 9 due, pass out Problem Set 10 (10a is through R-88N, 10b starts at R-28A)** **Solve Problem Set 9**
12	M Apr 10 W Apr 12 F Apr 14	**Pavia** Chapter 8 Lambert Ch. 13-15 **Solve Problem Set 10a** or Guest Lecturer (Landis) **Solve Problem Set 10b**	Reich handout 3	Introduction to Mass spectrometry Presentation of data and nomenclature Isotopes, molecular formulas, High and low resolution techniques Different ionization techniques Analysis of ions, Different types of detectors Analyzing fragmentation patterns **Solve Problem Set 10a** **written report on unknowns due** **Solve Problem Set 10b during class, then turn in entire PS 10**

		or Guest Lecturer (Landis)		
13	M Apr 17	Dr. Guzei hand-out	----------	Dr. Ilia Guzei, guest lecturer
	W Apr 19	Dr. Bates hand-out	----------	Dr. Desiree Bates, guest lecturer Computational methods in NMR
	F Apr 21	Dr. Martha Vestling guest lecture on Mass Spectrometry	----------	Dr. Martha Vestling guest lecture on Mass Spectrometry Modern mass spec techniques Capabilities of the UW-Mass Spec Center
14	M Apr 24	Class presentations	----------	Class presentations
	W Apr 26	Class presentations	----------	Class presentations
	F Apr 28	Class presentations	----------	Class presentations
15	M May 1	Class presentations	----------	Class presentations
	W May 3	Class presentations	----------	Class presentations
				Final unknown report is due the day of the final exam, which we will decide on later in the semester.