

Chemistry 425/395
Spectroscopy and Structural Elucidation

Dr. David Crumrine
Office: FH 212
Phone: 773-508-3114
Fax: 773-508-3086
email: dcrumri@luc.edu

Spring 2010
MW 5:30-6:50, FH 105

This course will introduce the use of spectroscopy for the elucidation of organic structures. The methods will include IR spectroscopy, UV-Vis spectroscopy, Mass spectrometry, NMR techniques (^1H , ^{13}C , and 2D), X-ray, and related computational techniques. The course will focus on the application of these methods in solving structures of organic molecules, with some instrument theory, some history, and method development.

Course Information:

1. Grading:	Midterm Exam 1	100pts	22.2%
	Midterm Exam 2	100pts	22.2%
	Final Exam	150pts	33.3%
	Problem Sets #1-3	75pts	16.7%
	Presentation	25pts	5.6%
	Total	450pts	100%

2. **Office Hours:** MW 4:30- to before class in FH 212; other times, by appointment.

3. **Textbook** P. Crews, J. Rodriguez, M. Jaspars, "Organic Structure Analysis 2nd Ed," Oxford Univ. Press, 2009.
This text is the only source material permitted during exams.

4. Other General References Spectroscopy Texts:

Lambert, Shurve, Lightner, Cooks, "Organic Structural Spectroscopy", Prentice-Hall, Upper Saddle River, NJ, 1998.
Silverstein, Webster, Kiemie "Spectroscopic Identification of Organic Compounds, 6th Ed. Wiley 2005.
Pavia, Lampman, & Kriz "Introduction to Spectroscopy 3rd Ed" Saunders College Pub, 2001.
Williams and Fleming, "Spectroscopic Methods in Organic Chemistry" 5th Ed. McGrawHill, 1995
Field, Sternhell, Kalman, Organic Structures from Spectra 4th Ed., Wiley, 2008

5. Other Spectroscopy Texts:

Breitmaier, "Structure Elucidation by NMR in Organic Chemistry"
Derome, "Modern NMR Techniques for Chemistry Research" Pergamon, 1987.
Duddeck, "Structure Elucidation by Modern NMR"
Jacobsen, "NMR Spectroscopy Explained" Wiley, 2007.
Kemp, "Organic Spectroscopy" 3rd Ed. Freeman, NY 1991.
Macomber, "A Complete Introduction to Modern NMR Spectroscopy," Wiley, 1998.
McLafferty & Turecek, "Interp. of Mass Spectra" 4th Ed", University Science Books, 1993.
Nelson, J. H., "NMR Spectroscopy" Prentice Hall, NJ, 2003.
Pretsch, Buhlmann, Affolter, "Structure Det. of Organic Compds 3rd Ed." Springer, 2000
Wehrli, Marchand, & Wehrli "Interp. of Carbon-13 NMR Spectra" 2nd Ed, Wiley, 1988.

6. Computational Suites

ACD Labs, Hyperchem, ChemDraw Professional
ChemWindows-Spectroscopy & newer versions

7. Schedule

Spectroscopy Chemistry 425-/395, Spring 2010

Lecture Outline

(Tentative)

Date	Chap	Topic	Tentative Lecturers
Jan 20	1	Background Info: Introduction; Analysis or Separation of mixtures; Purification.	DC
Jan 25	2/3	¹ H NMR: History, definitions, theory, chemical shifts, assignments, integration	JB
Jan 27	4	¹ H NMR: coupling constants, signs, classification of spin systems, problems.	JB
Feb 1	2	NMR: relaxation (T ₁ & T ₂), simulations, solvent effects, problems	JB
Feb 3	3/4	¹³ C NMR: Theory, Chemical shifts, Coupling, Decoupling, nOe, Assignments,	DC
Feb 8	5-5.3	¹³ C NMR: APT, Relaxation, INEPT, DEPT techniques	DC
Feb 10		NMR: Computations, Simulations, Problem Solving Problem Set #2	DC
Feb 15	9	IR: Theory of Dispersive & FTIR, characteristic absorptions, symmetry	JC
Feb 17	9	IR: absorptions cont 'd, problem solving, databases, Raman, AFM, SEM	JC
Feb 22	10	UV-Vis: Theory, excited states, transitions, chromophores, Woodward-Fieser rules	DC
Feb 24	10	UV-Vis:, CD, ORD, <i>etc.</i> Diode-array LC detectors.	DC
Mar 1		Problem Solving combining NMR, IR, and UV/Vis	DC
Mar 3		Midterm Exam #1	
Mar 8-10		<i>Mid-semester break</i>	
Mar 15	1 pg	27EPR, Electronic relaxation, ENDOR	RH
Mar 17	2	More NMR: VT, Heteroatoms, CIDNP, Solids Problem Set #3 due	DF
Mar 22	11	ACS2DNMR: Introduction, Theory, COSY, TOCSY, HETCOR, nOesy	DF
Mar 24	11	ACS2D NMR: Techniques, indirect detection, HMQC, Acronyms, & Applications	DF
Mar 29		2D NMR: Problem solving	DF
Mar 31		X-Ray Diffraction	DL
Apr 5	6	MS: Theory, Instrumentation, and Combined Techniques	DC
Apr 7	7	MS: Analysis of small and large molecules	DC
Apr 12	8	MS: Fragmentation processes in e- ionization MS, problem solving	DC
Apr 14		Midterm Exam #2	
Apr 19	15	Larger Molecules & Other Techniques	
Apr 21		<i>Student Presentations</i>	
Apr 26		<i>Student Presentations</i>	
Apr 28		Review Day & Problem Solving practice	
May 2?		Final Exam	

Lecturers: J.Babler, J.Ciszek, D.French, R.Holz, D.Liu

8. Spectroscopy Websites that may be useful (Spring 2010). Using Google: "Spectroscopy" gave 1.9×10^7

hits; "Organic Spectroscopy" gave 5×10^6 hits; "Spectroscopy Problems" gave 3.5×10^6 hits; etc.

Twelve Examples are listed below.

1. en.wikipedia.org/wiki/Spectroscopy The first listing from "Spectroscopy," lots of info and branches.

2. spectroscopyNOW.com - spectroscopy and spectrometry portal

Spectroscopy portal addressing mass spectrometry, NMR, MRI, x-ray, atomic, Raman, IR, UV, proteomics and chemometrics and informatics techniques. You can register for info. www.spectroscopynow.com

3. [WebSpectra - Problems in NMR and IR Spectroscopy](#)

More NMR practice problems and a great outline of spectral assignments methods.
www.chem.ucla.edu/~webspectra/ - 21k. mainly ^1H and ^{13}C only a few others.

4. [Spectroscopy Home](#) Problem Sets: Infrared **Spectroscopy** Problem Set: ^1H NMR Problem Set: ^{13}C NMR Problem Set · Mass **Spectroscopy** Problem Set · Integrated **Spectroscopy** Problems ...
www.chem.uic.edu/web1/OCOL-II/WIN/SPEC.HTM - 3k

5. [Organic Chemistry On Line](#) A good introduction to modern NMR **spectroscopy**. ... A nice collection of problems using all the **spectroscopy** methods discussed here. ...
www.cem.msu.edu/~reusch/VirtualText/Spectrpy/spectro.htm - <http://www.cis.rit.edu/htbooks/nmr/> {exceptional}

6. [NMR Spectroscopy - Theory](#) A nice little intro to NMR **spectroscopy** theory.
teaching.shu.ac.uk/hwb/chemistry/tutorials/molspec/nmr1.htm

7. [Spectroscopy](#) **Spectroscopic** databases can aid the chemist in spectral interpretation and structure elucidation. Searches can be conducted by inputting then cds.dl.ac.uk/cds/datasets/spec/specinfo/spectro.html

8. [Organic Structure Elucidation Workbook](#) <http://www.nd.edu/~smithgrp/structure/workbook.html> Good Problems ^1H , ^{13}C and MS with relative difficulty. No answers included.

9. [Spectroscopy Problems](#) We have used these **problems** for many years in the **spectroscopy** section of the **organic** chemistry lab and lecture courses. orgchem.colorado.edu/hndbksupport/specttutor/main.html - 6k Problems include ^1H NMR and IR data with answers and some interpretation.

10. [CHP - Spectroscopy](#) *Spectroscopy* is the use of the absorption, emission, or scattering of electromagnetic radiation by matter to qualitatively or quantitatively study the ...
www.files.chem.vt.edu/chem-ed/spec/spectros.html - [Cached](#) - [Similar](#)

11. http://www.aist.go.jp/RIODB/SDBS/cgi-bin/cre_index.cgi Japanese Institute website with combined spectra. Used for early problem set.

12. http://nmrsg1.chem.indiana.edu/other_sites.html A long list of NMR related websites.