CHEMISTRY 528 SYLLABUS Spring 2008
Contemporary Methods of Synthesis & Analysis I
Lecture: MW NS110 Lab: Various times

INSTRUCTORS:

Mass Spectrometry: Dr. Richard M. Higashi, BRB 347, phone: 852-7496, rick.higashi@louisville.edu

Organic Chemistry: Dr. Syed R. Hussaini, C.B. 334, phone: 852-7499, srhuss01@louisville.edu

2D NMR: Dr. Teresa W-M. Fan, BRB 348, phone 852-6448, twfan001@gwise.louisville.edu

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GTA: Mr. Jose Carlos Aponte Silva, jcapon01@louisville.edu

PREREQUISITES: Satisfactory completion of Chem 527 (Introduction to Separations and Spectroscopy)

TEXTBOOK: **Organic Structural Spectroscopy (1998 edition) by J. B. Lambert, H. F. Shurwell, D. A. Lightner and R. G. Cooks**. Lectures will depart from the textbook, but reading the assigned sections is strongly recommended for better course performance, and purchasing the textbook may aid your professional development.

OTHER: Special supplements from the Instructors as needed.

OVERVIEW: This course is based on an innovative course series originated by Drs. Yappert and Gibson, to provide realistic experiences that highlight the integral nature of chemical synthesis and analytical characterization. With this purpose in mind, the lecture component of the course will cover principles, theoretical, and experimental concepts on a variety of powerful contemporary approaches to chemical synthesis and characterization methods. Selected syntheses and applications will be demonstrated in the laboratory and the factors that influence the choice of analytical measurement will be highlighted. The specific suggested readings are identified below.

Section A: Nuclear Magnetic Resonance Spectroscopy and its Applications

1) **Weeks 1-4;** Special supplements, Chap. 2, 3, 4, 5.1-3, 6.1, 6.2 in text: review of basic principles of ^{1}H and ^{13}C NMR; introduction to the acquisition and processing of NMR data; NMR analysis and structural assignments of terpene or other unknowns by 1D and 2D NMR (COSY, HETCoR).

2) **Weeks 5-6;** Special supplements, Chap. 5.5, 6.3 in text: Hetero Diels-Alder reaction and structural analysis of the products by 2D NMR (NOESY).

3) **Weeks 7-8;** Special supplements, Chap. 10.3 in text, Organic survey text: Enzymatic resolution of menthyl esters, analysis by ^{13}C NMR, polarimetry.

Section B: Mass Spectroscopy and its Applications

4) **Weeks 8-11:** Chap. 13, 14 in text: Introduction to GC-MS; characteristic fragmentation patterns of organic compounds; identification of chlorinated and brominated compounds in a mixture; introduction to MALDI-ToF-MS.

5) **Weeks 13-end:** Special supplements: Synthesis of a polymer by ring opening metathesis polymerization (ROMP), analysis of the polymer by NMR spectroscopy and MALDI-ToF-MS.

EXAMINATIONS AND REPORTS:

There will be two examinations each at the end of sections A (150+150 = 300 pts.) and B (100+100 = 200 pts.). Also, there will be five laboratory reports worth 500 points (total). Upon completion of each experiment (e.g. first one will take 4 weeks), each report is due in to the GTA **prior** to the start of the next lab session. Late reports will be deducted 25% per week, from the total possible points. For example, if you are 10 min late on Report #A.1, you can get a maximum of only 112.5 pts.

Tentative exam dates: #1 (based on Section A), Mon., Feb. 18 and Wed., Feb. 20
#2 (based on Section B), Wed., Apr. 16 and Mon., Apr. 21

Lab Repeat or Make-up Policy: There will be no lab experiment **repeats**. If a student is absent for one of the labs, he/she is required to present a legitimate, documented excuse (e.g. letter from doctor) for the missed lab, whereupon the instructors will decide on the nature and time of the **make-up** experiment and report. In any case, total of only one lab can be a **make-up** to retain full credit. For additional information regarding absence due to University events, see http://louisville.edu/provost/what-we-do/classroom-policies/classroom-policies.html

Literature Research Assignment for Undergraduates: In subsequent weeks, the instructors will announce these extra credit assignments, tentatively worth 50 pts each.

Graduate Students: The Literature Research assignments are mandatory.

Tentative Distribution of Lab Report Points

Report #	Topic	Points
A.1	2D NMR/terpenes	150 (120 Org + 30 Analyt)
A.2	Hetero Diels Alder/NOESY	80 (60 Org + 20 Analyt)
A.3	Enzymatic resolution of menthyl esters Application of Polarimetry	40 (Org) 40 (Analyt)
B.1	GC/MS Extraction of volatile compounds Identific. of chlor./brom. Compounds	40 (Analyt) 40 (Analyt)
B.2	ROMP and analysis MALDI and PSD/MALDI	50 (Org) 60 (Analyt)
	Sub-Total Organic	**270**
	Sub-Total Analyt.	**230**
	TOTAL	**500**

GRADING:

Your grade will be established from the percentage of the 1000 points that you have accumulated; the grade scale (as percentages) will be as follows:

90-100 = A's
80-89 = B's
70-79 = C's
60-69 = D's
< 60 = F

Graduate Students: The Literature Research assignments are mandatory and thus will be included in the points total. Therefore, Graduate Students are graded based on the percentage of 1200 points.

The instructors reserve the right to make changes in the syllabus when necessary to meet learning objectives, to compensate for missed classes, or for similar reasons.

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Tentative Schedule for Spring 2008

Week #1	**Lecture:** NMR, review of fundamental principles of FT-NMR (RMH[1])	M (01/07/08)
	Lecture: Introduction of 2D NMR applications to characterization of organic compounds (SRH[1])	W (01/09/08)
	Lab: 1D NMR characterization of terpenes, lab exercises	
Week #2	**Lecture:** 2D NMR: principles of COSY and TOCSY (TWF[1])	M (01/14/08)
	Lecture: Applications of 2D NMR: COSY (SRH[2])	W (01/16/08)
	Lab: COSY characterization of terpenes	
Week #3	No lecture (Martin Luther King Jr. Day)	M (01/21/08)
	Lecture: Applications of 2D NMR: COSY (SRH[3])	W (01/23/08)
	Lab: HETCoR of terpene unknowns	
Week #4	**Lecture**: 2D NMR: principles of HETCoR and pulse sequences (TWF[2])	M (01/28/08)
	Lecture: Applications of 2D NMR: HETCoR (SRH[4])	W (01/30/08)
	Lab: Finish COSY and HETCoR of terpene unknowns on 500 MHz NMR. Effect of exp. variables on T1 and T2	
Week #5	**Lecture:** NOE, fundamental concepts; homonuclear, heteronuclear enhancement, effect of τ_c on NOE (TWF[3])	W (02/04/08)
	Lecture: 2D NMR Advanced Applications (TWF[4])	W (02/06/08)
	Lab: Hetero Diels Alder; analysis by ^{1}H NMR and NOESY	
Week #6	**Lecture**: Diels-Alder reactions/NOESY (SRH[5])	M (02/11/08)
	Lecture: Chirality, polarimetry, ORD and CD (ANL[1])	W (02/13/08)
	Lab: Hetero Diels Alder; analysis by ^{1}H NMR and NOESY	

Week #7	**Lecture:** EXAM I (Analytical Chem.)	M (02/18/08)
	Lecture: EXAM I (Organic Chem.)	W (02/20/08)
	Lab: First two hours: Quantitative applications of polarimetry: calibration and determination of enantiomeric excess in mixtures of (+) and (-) menthol Last two hours: start enzymatic resolution of menthyl esters	
Week #8	**Lecture:** Kinetic resolution of enantiomers (SRH[6])	M (02/25/08)
	Lecture: Intro to GC-MS (RMH[2])	W (02/27/08)
	Lab: Completion of identification of menthyl ester product by ^{1}H and ^{13}C NMR and polarimetry	
Week #9	**Lecture**: MS fragmentation (SRH[7])	M (03/03/08)
	Lecture: MS fragmentation – modern tools & implications (RMH[3])	W (03/05/08)
	Lab: Analysis of volatile (fragrant) compounds by GC/MS	
Week #10	**Spring Break** (03/10/08 to 03/16/08)	
Week #11	**Lecture:** GC-MS (cont), Basics of MS, other types of MS (RMH[4])	M (03/17/08)
	Lecture: LC-MS (RMH[5])	W (03/19/08)
	Lab: Analysis of volatile (fragrant) compounds by GC/MS	
Week #12	**Lecture**: How Synthesis, Chromatography, and MS work together (RMH[6])	M (03/24/08)
	Lecture: MALDI-ToF MS: principles, instrumentation (RMH[7])	W (03/26/08)
	Lab: Identification of chlorinated and brominated compounds in mixture (based on fragmentation)	
Week #13	**Lecture:** Ring-opening metathesis polymerization (SRH[8])	M (03/31/08)
	Lecture: Ring-opening metathesis polymerization (SRH[9])	W (04/02/08)

	Lab: First two hours: start ROMP experiment Last two hours: demonstrate applications of MALDI with small peptides and larger molecules (using different matrices)	
Week #14	**Lecture**: Inorganic MS & the Future (RMH^{8})	M (04/07/08)
	Lecture: Review for Exam II (Organic Chem.) (SRH^{10})	W (04/09/08)
	Lab: First two hours: continuation of ROMP Last two hours: PSD in MALDI: principles, applications	
Week #15	**Lecture**: Review for Exam II (Analytical Chem.) (RMH^{9})	M (04/14/08)
	Lecture: Exam II (Organic Chem.)	W (04/16/08)
	Lab: Completion of ROMP, analysis by MALDI and NMR. Checkout and evaluation	
	Last Lecture: Exam II (Analytical Chem.)	M (04/21/08)

SRH = 10 lectures
RMH = 9 lectures
TWF = 4 lectures
ANL = 1 lecture
Total = 24 lectures + 4 exam sessions