

CHEMISTRY 455/555

ADVANCED NMR TECHNIQUES

Winter 2006

Instructor: Dr. George S. Kriz

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Office Hours: Monday and Friday 10:00 to 12:00; Monday 3:00 to 5:00

If you wish to see me at other times, you are encouraged to do so. All I ask is that I may be notified in advance, in order to ensure that I will be available.

Course Prerequisites: The prerequisite for this course is a passing grade in Chemistry 454 or 554. Students who do not have this prerequisite should consult with me as soon as possible.

Textbook: The *recommended* textbook is:

Timothy D. W. Claridge, *High-Resolution NMR Techniques in Organic Chemistry*. Tetrahedron Organic Chemistry Series Volume 19, Elsevier, Amsterdam: 1999.

Lecture: Chemistry Building 485, Tuesday and Thursday at 11:00

Laboratory: There is no scheduled time for the laboratory. Each student will work on the Varian Inova 500 MHz NMR instrument according to their own convenience.

Laboratory Safety Examination: In order to be enrolled in this course, each student will be required to take *and pass* a laboratory safety quiz. This organic chemistry safety quiz is an on-line quiz, consisting of 25 multiple choice questions. It is your responsibility to take and pass the safety quiz on your own time, during its scheduled times, before Thursday, January 12 at 12:00 noon. If you cannot successfully pass the quiz, you will fail or be dropped from this course.

The required passing score on the quiz starts at 80% and increases as the final deadline approaches.

Quiz Times:

1st Try: Wednesday, January 4, 8:00 AM – Friday, January 8, 5:00 PM (80% is passing)

2nd Try: Friday, January 6, 5:30 PM – Tuesday, January 10, 5:00 PM (84% is passing)

3rd Try: Tuesday, January 10, 5:30 PM – Thursday, January 12, 12:00 noon (88% is passing).

ASAP go to <http://argon.chem.wvu.edu/OCQLink.htm> for more information. You are strongly encouraged to review the safety information before taking the quiz the first time. You must pass the quiz by the above deadlines, or you will not be allowed to proceed further in the course.

If you cannot successfully pass the quiz, you will fail or be dropped from this course.

The quiz can be taken using the Chemistry Department Computer lab located in CB 280 for from any personal computer with internet access. Dial-up access from home might be slower.

The safety quiz will cover material that is provided in the following textbook chapters:

D. L. Pavia, G. M. Lampman, G. S. Kriz, and R. G. Engel, *Introduction to Organic Laboratory Techniques: A Microscale Approach (Third Edition)*, Saunders College Publishing, Philadelphia: 1999.

Laboratory Safety	pp. 4 through 20
Heating and Cooling Methods	pp. 521 through 528
Reaction Methods	pp. 529 through 530

A complete review of the material covered by the safety quiz is available on the Chemistry Department web page. The URL address for this safety review is:

<http://atom.chem.wvu.edu/dept/osafety/index.htm>

A complete instruction page for the organic chemistry safety quiz may be found at:

<http://argon.chem.wvu.edu/OCQLink.htm>

A link to the actual safety quiz is included at the bottom of the instruction page.

Examinations: There are no examinations planned for this course.

Grading Standards: The grades in this course will be based on five laboratory reports, each of which has a value of 50 points. The laboratory reports will be graded on completeness and clarity of the presentation of the experimental results and discussion, and they will also be graded on the form of the presentation (format, correct use of English grammar, correct syntax, etc.).

NOTE: Students who enroll in Chemistry 555 will be required to complete a sixth laboratory experiment. The total points for Chemistry 555 students will be adjusted to correspond to the grading scale shown below.

Grades will be assigned on an absolute scale of points according to the following standards:

A	225 to 250 points (90 to 100%)
B	200 to 224 (80 to 89%)
C	163 to 199 (65 to 79%)
D	138 to 162 (55 to 64%)
F	0 to 137 (0 to 54%)

A grade of Incomplete (K) may be assigned if students are unable to complete the assigned work for the course, provided that (1) the instructor is notified in advance, (2) the student is doing passing work at the time that a request for a "K" grade is made, and (3) an Incomplete Grading Contract is completed before the final course grades are due. Students are advised that according to University policy, after one year, if the "K" grade has not been removed, it will automatically revert to a failing ("Z") grade.

Laboratory Fee: A laboratory fee of \$26.36 has been included as a part of the registration fees that each student must pay in order to enroll in this course. This laboratory fee is intended to offset the costs associated with operating and maintaining the NMR instrument and its software. Students who are responsible for an excessive amount of damage to the instrument or its ancillary components will be required to pay for these costs to the extent that they exceed the laboratory fee.

SCHEDULE OF CLASS MEETINGS

<u>DATE</u>	<u>TOPIC</u>
January 5	ORIENTATION Introduction to Fourier Transforms
10	Fourier Transforms; Introduction to Pulse Sequences

	12	Introduction to Pulse Sequences; Magnetization Vectors
	17	Generation of NMR Signals
	19	Relaxation Processes; Pulse Sequences
	24	Free Induction Decay; Double Resonance
	26	The Nuclear Overhauser Effect
	31	Gated Decoupling; Off-Resonance Decoupling
February	2	Measurement of Relaxation Times
	7	The Spin-Echo Experiment; The Attached Proton Test
	9	Polarization Transfer; The INEPT Experiment
	14	The DEPT Experiment
	16	Introduction to Two-Dimensional NMR; J Spectroscopy
	21	J Spectroscopy; The COSY Experiment
	23	The COSY Experiment
	28	The HETCOR Experiment
March	2	The HETCOR Experiment
	7	The INADEQUATE Experiment
	9	The NOESY Experiment and Magnetic Resonance Imaging
	14	CLASS EVALUATION (9:00 AM)

LIST OF EXPERIMENTS

1. NMR Spectroscopy of Camphor
Each student works *individually*
50 Points
2. Nuclear Overhauser Difference Spectroscopy
Students work in teams
50 Points
3. Spin Decoupling and COSY Spectroscopy
Students work in teams
50 Points
4. gHETCOR and gHSQC Spectroscopy
Students work in teams
50 Points
5. 2D-Nuclear Overhauser Enhancement Spectroscopy (NOESY)
Students work in teams
50 Points

STUDENTS ENROLLED IN CHEM. 555, ONLY:

6. gHMBC Spectroscopy
Graduate students will work in teams
50 Points

For each of the experiments, a standard sample in a sealed NMR tube will be prepared and available. A specific instruction sheet for each experiment will also be available upon request. Each of the experiments will be conducted using the Varian Inova 500 MHz NMR instrument. In case of exceptionally heavy traffic on the 500 MHz instrument, your instructor may direct you to use the Varian Mercury 300 MHz instrument, instead.

STATEMENT REGARDING THE GROUP PROCESS: For the group assignments, each student will be assigned a specific role. The assignment and the specific guidelines for each role will be included in the assignment sheet prepared for each specific experiment. Note clearly, however, that, regardless of the assigned role, each student shares in the responsibility for the completion of the assignment and for the correctness of the material included in the report.