

Chemistry 4406/6406
Molecular Spectroscopy Laboratory Information and Guidelines 2017

Prep lab (203a), Instrument lab (317a) NMR lab (027b)

Tuesday 1-4 PM

<http://www.memphis.edu/chem/faculty-burkey/tjb-4416.php>

Instructors: Sara Brady, Scott Powers, Smith 203, (Office hours by arrangement) x4354

Supervisor: Ted Burkey, Smith 205 (CCLC Friday 11 AM), x2634

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Policy of Equal Access

Students who require academic adjustments in the classroom due to a disability must first register with the University of Memphis Disability Services. Following registration and within the first two weeks of class, please contact supervisor to discuss appropriate academic accommodations. Appropriate arrangements can be made to ensure equal access to this course.

Textbooks:

Spectrometric Identification of Organic Compounds”, Silverstein, Webster, Kiemle 7th Ed.

Grading: Sum of all lab report grades

An approximate grading scale follows that equates the percentage scale to the U of Memphis grading system.

90-100 = A

80-89 = B

70-79 = C

60-69 = D

<60 = F

4406/6406 Laboratory Information:

Laboratory Attendance:

Attending lab is mandatory. If a situation arises such that a student misses a lab, please contact your instructors as soon as possible (preferably by email). The student must arrange within two weeks with an instructor to make up the missed experiment. An excused absence must be documented (examples include: death in the family or documented sickness). Any absence deemed excused by the University of Memphis will be considered acceptable. It is the student's responsibility to document an absence as excused and to schedule an appropriate make-up date.

Note: Since much of this course involves instruction which cannot be obtained from a textbook, unexcused absences will result in a grade of zero on any experiment missed. It is the responsibility of the student to arrange to make up lab for an excused absence.

Laboratory Safety:

Students are required to wear eye protection at all times during the laboratory. We are not allowed to provide you with eye protection—you must provide your own. There will be no horseplay. No more than three warnings will be given to a student to adhere to the rules of a safe laboratory. A student who do not use eye protection, dress or behave in an unsafe manner will be asked to leave the laboratory. Appropriate clothing will be worn in the laboratory. Open toed shoes (sandals and “flip-flops) are not acceptable. Shorts and dresses should be avoided and if they are worn, an apron or lab coat (student's responsibility) should be used. We are not responsible for any damage to your clothing—so dress appropriately for the lab—older “work” clothes are best. Students will be given a review of safety rules and sign a sheet to acknowledge that they have read and understand the safety rules.

Lab Notebooks: A hardbound laboratory notebook will be used to record all experimental procedures and data. This may be discussed and reviewed periodically.

Lab reports

A report will be prepared for each experiment and is due one or two weeks (as announced) after the experiment. Late lab reports will be docked 5 % for each day late. The report must be a Word document and delivered by email. Instrument spectral files should be attached. Processed spectra must be integrated at the end of the Word document. After evaluation by the instructor, revisions may be required. There will be two types of lab reports: one for compounds of known structure and one for unknown structure. An example grading guide will be handed out.

Instrument Reservations and Software

Instruments must be reserved on FACES before use. You must submit an application to use FACES ([FACES/Instrument use Application](#)) and to use instruments you must adhere to all instrument guidelines ([NMR Guidelines](#)). You may reserve an instrument other than regular 4406 hours: Tuesday 1-4 pm reservations have been made for 4406: GCMS (Jan 24, 31, Feb 7) NMR spectrometer (March 14, 21, 28, and April 4). You will need to borrow key to access Smith Chemistry 027b. Structure drawing software can be obtained at <http://sitelicense.cambridgesoft.com/sitelicense.cfm?sid=1161>. NMR spectra processing software can be obtained from JEOL: go to nmrsupport.jeol.com and create a JEOL software user account, login to your software user account, download the JEOL Delta software and install it. You will need to obtain a Machine ID (for data processing only) and send it to Dr. Burkey so he can obtain a license key for you. You will need to know computer type, operating system and version including bits. To obtain a machine ID, start Delta software, select OPTION>LISCENCE KEY. Return to nmrsupport.jeol.com and select Obtain a License Key. Alternatively, you can use the auxiliary computer next to the JEOL computer to process your data without reserving the spectrometer.

Type I – Instrument function and use and known sample analysis 100 pts total

The report will include the followings sections (approximate pts, depends on experiment):

- I. Introduction and purpose. (15 pts)
- II. Block diagram of instrumentation (cannot be copied, thus your own creation) and a description of how the instrument functions. (15 pts)
- III. Theory: why the analyte gives a signal; where the analyte signal arises from. (10 pts)
- IV. The experimental procedure (25 pts)
- V. The Results (25 pts) including the following
 - Copies of spectra and or plots
 - Tabulate spectra information: peak positions (masses, frequency etc) intensities or areas, other features (multiplicity), peak assignment (functional group, fragment formula, etc)
 - Other observations
- VI. Discussion and Conclusions (10 pts)
 - Show structure of compound and label it with relevant peak positions etc.
 - Explain why conditions affect spectral features

Type II – Analysis of unknown samples 100 pts total

- I. Introduction and purpose. (10 pts)
- II. The experimental procedure (30 pts)
- V. The Results (30 pts) including the following
 - Copies of spectra
 - Tabulate spectra information: peak positions (masses, frequency etc) intensities or areas, other features (multiplicity), peak assignment (functional group, fragment formula, etc)
 - Other observations
- VI. Discussion and Conclusions (25 pts)
 - Show structure of compound and label it with relevant peak positions etc.
 - Show structures of all classes of possible compounds if there is not enough data for exclusive determination
 - Explain basis of assignment (in NMR – area, coupling, etc, in MS difference in masses, exact masses, etc)

Each laboratory experiment must be turned in for grading during the next laboratory period. If a laboratory is turned in late, there is a penalty of 5 percentage points per day. After 20 academic days the grade is 0.