CHM 426/526

Spectroscopic Identification of Structure

Spring 2019

MWF 8:30am–9:25am, 158 Hughes Hall

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Office Hours
Tu, Th 2:00pm–3:00pm
(*Please feel free to drop by at any time.*)

Course Objectives. CHM 426/526 is designed to develop your ability to use spectroscopic information to solve organic structural problems. Specifically, we will focus on the use of mass spectrometry, infrared spectroscopy, ^{1}H and ^{13}C NMR spectroscopy, and two-dimensional NMR methods. The theory behind each analytical technique will be introduced; however, *the emphasis of this course is practical interpretation of spectroscopic data*. To succeed you will need to do many problems. Significant in-class time will be devoted to problem solving.

Texts for CHM 426/526: The textbook for this course is *Spectrometric Identification of Organic Compounds*, 8th Ed., by Silverstein, Webster, and Kiemle. *Organic Chemistry* by David Klein, and *Organic Chemistry*, 5th or 6th ed., by Paula Bruice may be helpful, but are not required.

Additional Required Materials:
Molecular model kit. Access to Canvas and CHM426/526 websites for assignments, lecture slides, etc.
ChemDraw: We have a site license for this very useful program. You will be given instructions on how to download this program to your laptop.
Clicker: Turning Point Response Card NXT or smart phone Response app is required. Please be sure to bring your clicker to every class. Participation in class is very important. Questions to probe your understanding will be presented during lectures via clicker, which allows you to participate anonymously. Instructions on how to register your clicker can be found on Canvas.

Grading. Grades in this course will be derived from a combination of in class quizzes, and take-home midterm and final exams. Take-home exams are exams, and as such you should work alone. A rubric for grading the quizzes and exams in this course can be found on Canvas and the CHM426/526 website. Grades awarded to answers written in pencil will not be changed after tests or assignments have been returned.

Quizzes (10):	200 (missed quiz may be made up with **identifying one unknown**)
Midterm:	50
Final:	50
Course Total:	300

Approximate cutoffs: A ≥ 90%, B ≥ 75%, C ≥ 60%, D ≥ 50%, F < 50%. Plus/minus grades will be awarded. **Graduate students enrolled in CHM526 will be required to present one structure problem from recent literature**.

Tentative Schedule

Week	Date	Subject	Exercises
1	01/28-30	Organic compound structures	Review of
		Functional and Stereoisomers (quiz 1)	O-Chem.
2	02/1-6	MS: The molecular ion, index of unsaturation	MS problems
		IR: Theory and instrumentation	IR problems
3	02/8-13	^{1}H NMR: Introduction, theory	Combi problems
		Quiz #2 (02/11, IR & MS)	(Beginner)
		^{1}H NMR: Chemical shifts, chemical equivalence	Combi problems
4	02/15-20	^{1}H NMR: *J* coupling, 1st order spectra	(Beginner)
		Quiz #3 (02/20, IR, MS, and ^{1}H NMR)	Combi problems
5	02/22-27	^{13}C NMR: Intro, theory	(Beginner)
		^{13}C NMR: Chemical shifts	
6	03/1-6	Quiz #4 (03/01, IR, MS, and ^{1}H & ^{13}C NMR)	
		^{1}H NMR: 2nd order effects, magnetic equivalence	Combi problems
		^{1}H NMR: Spin systems, equivalent protons	(Intermediate)
7	03/8-13	^{13}C NMR: DEPT, signal intensities	Combi problems
		Quiz #5 (03/11, IR, MS, and ^{1}H & ^{13}C NMR)	
8	03/15-20	^{13}C NMR: ^{1}H–^{13}C *J* coupling, spin decoupling	
		Quiz #6 (03/18, IR, MS, and ^{1}H & ^{13}C NMR),	Midterm distributed (due 3/27)
9	03/25-31	Spring break	
10	04/1-4/5	MS: Interpretation, fragmentation	Combi problems
		IR: Interpretation, functional group correlations	(Intermediate)
11	04/8-12	Quiz #7 (04/08, IR, MS, and ^{1}H & ^{13}C NMR)	
		Population effects: the nuclear Overhauser effect	Dr. Ramelot, guest
12	04/15-19	Population effects: polarization transfer	lecturer (4/10)
		Quiz #8 (04/15, IR, MS, and ^{1}H & ^{13}C NMR)	
13	04/22-26	2D NMR: Intro, ^{1}H–^{1}H COSY	Combi problems
		Quiz #9 (04/22, IR, MS, ^{1}H & ^{13}C and COSY NMR)	(Challenging)
14	4/29-5/3	2D NMR: HSQC, HMBC, HMQC	
		2D NMR: 1H–1H COSY/TOCSY	
15	05/06-10	Quiz #10 (04/29, IR, MS, and ^{1}H & ^{13}C NMR and 2D NMR)	
		2D NMR: NOESY, ROESY	Combi problems
		Final exam distributed (due 5/15 by 5:00 pm)	(Expert)*

This section is for CHM526 (graduate students) only.

20 Minutes Presentation: Identification of An Unknown Using Organic Spectroscopy

Instruction

Modern spectroscopic techniques such as IR, Mass, and NMR are vital to the identification of the structure of an organic compound. Today IR spectroscopy makes it easy to identify the functional groups present in a molecule. NMR spectroscopy establishes the carbon-hydrogen framework of the molecule. Mass spectroscopy provides the molecular weight and whether halogen atoms such as chlorine or bromine are present in a molecule. A combination of these spectroscopic methods provides a powerful tool for the identification of the structure of new organic compounds. In this assignment, you are asked to present the spectroscopic evidence for one compound in a database of organic compounds. The databases that likely to contain such data include SDBS https://sdbs.db.aist.go.jp/sdbs/cgi-bin/cre_index.cgi

PubChem, https://pubchem.ncbi.nlm.nih.gov/

NCI/CADD, https://cactus.nci.nih.gov/chemical/structure

KnowItAnyWare, https://www.knowitallanyware.com/#login?id=search&type=un

SciFinder, https://www.lib.miamioh.edu/databases/S

The spectra are usually found in the property links to the compound.

Ph.D dissertations and Masters' thesis in organic synthesis can be found on line. They usually contain spectroscopic data.

Procedure

In this assignment, the following data should be collected.

- A quality (meaning readable by the class) IR spectrum of your chosen compound.
- A quality ^{1}H NMR spectrum of the same compound.
- A quality ^{13}C NMR spectrum of the same compound.
- A quality Mass Spectrum of the same compound.

In case where a spectrum is missing, the reported values in text should be presented. For example, if the mass spectrum is missing, the reported m/z peaks should be presented.

After you have collected all of the required data, prepare PowerPoint slides with each spectrum on a separate slide. Show your reference where you obtained the data. In your presentation, give detailed analysis for each set of data you display and their relationship to the compound structure. Merely give the compound structure is not acceptable. The structure should be labeled with numbers indicating each functional group giving rise to a specific signal in the spectra. Submit your presentation to your instructor via email attachment in a zip file after your presentation.

Alternatively, you may follow the instructions on the following page to obtain your own spectra and perform a presentation on how you solve an unknown structure.

Instructions on how to make up a quiz

In order to make up a missed/failed quiz, you may choose to identify an unknown organic compound. You have one week to do it starting from when you request for the unknown.

General steps to identify an unknown organic compound:

1. Obtain the unknown compound from your instructor;

2. Collecting data on modern instruments:

    (a) IR,

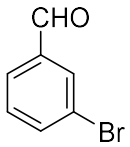
    (b) 1H NMR,

    (c) 13C NMR,

    (d) mass spectrum,

    (e) DEPT 13C and 2D NMR if needed.

3. Deduce the structure.



You will receive 100% worth of the quiz if you correctly identify the unknown and submit all spectra in good format.

Spectra: 50%, Structure: 50%