Chem 536 and 736 Spectroscopic Identification of Organic Compounds

Spring, 2021

Schedule Numbers: 20800 and 20806

*COURSE INFORMATION

Class Days: Tu/Th
Class Times: 5:00 – 6:15 PM

Class Location: https://SDSU.zoom.us/j/812 8523 1756
Lecture and discussion is online only

Instructor: Thomas Cole
tcole@sdsu.edu (preferred)
(619) 594-5579 (office)
Office Hours Location: CSL-210A (no visitors)
Office Hours Times (and by appointment):
M 8:00 – 9:00 AM
https://SDSU.zoom.us/j/86231401115
and Th 11:00 - 12:00 PM
https://SDSU.zoom.us/j/87538658767

Prerequisites

The prerequisites for those enrolling in Chem 536 is Chemistry 432, with a grade of C or higher. Also recommended is Chemistry 457 and 550 for undergraduates. Those students enrolling in Chem 736 do not have any prerequisites, since they are graduate students.

Enrollment Information

Please include information about enrollment for the course including, but not limited to:

- Students in this class are expected to have covered material found in Chem 232/432, 410A, 457 and 550. It is also assumed that a basic knowledge of spectroscopic methods, UV-Vis, IR, NMR (proton and carbon) and Mass Spectrometry is known. Experience in a research laboratory is also important to better understand the goals of this class.
- Drop deadline: 2/2/2021 at 19:59 (7:59 PM)

Concepts and Scope

Course Objectives:
This class intensively covers modern spectroscopic techniques used to identify and confirm structures of organic products. Problem solving and interpretation of spectra are strongly emphasized in addition to methods used in establishing purity of compounds. This course is based on ACS guidelines for characterization of small molecule suitable for publication and patents.

The purpose and scope of the course including, but not limited to:

- Spectroscopic methods used in the structural determination of organic compounds, establishment of purity and in some cases percentage yields.
- This classes covers the following topics:

Part 1 General Protocol to Solving Organic Spectral Problems
Characterization of Organic Compounds for Publication in ACS journals
Methods to determine empirical and molecular formulas

Part 2 UV-Vis
UV-Vis spectroscopy and determination of absorption maximum. Use of empirical rules for common functional groups

Part 3 Infrared Spectroscopy
Identification of the major organic functional groups.

Part 4 NMR

Basics of NMR, instrumentation, active nuclei,

Proton NMR, prediction of chemical shifts, spin-spin first and second order couplings and interpretation of proton NMR

Carbon-13 NMR, prediction of chemical shifts and interpretation of carbon NMR

Other nuclei such as: ^{31}P, ^{19}F and ^{11}B

2-D NMR, homonuclear and heteronuclear NMR

Part 5 Mass Spectroscopy

Low resolution and high resolution.

Different ionizations, separation of ions, MS/MS and detection.

Part 6 Quantification

Determination of yields, purity, enantiomeric and diasteromeric purities

Please note, there will be no classes or office hours on Rest & Recovery Days: Monday March 8, Tuesday March 30 and Thursday April 15. In addition, campus is closed on César Chávez Day, Wednesday March 31.

Student Learning Outcomes:

1. Students will be come effective at using infrared spectroscopy to characterize organic and organometallic compounds as well as identify unknown compounds.
   - The fundamental basics of infrared spectroscopy are covered in lecture and supplemented with supporting information , data sheets and examples on blackboard. In class worked problems are used to help develop a systemic approach to understanding interactions between structure and absorption bands that can be used to confirm organic products or identify unknowns.
2. Student will be able to use the basics of proton and carbon NMR for the interpretation of NMR spectra, extracting chemical shifts, coupling constants and integration values to characterize organic compounds and identify unknown compounds. They will also be able to used 2D NMR experiments for the full characterizations of these compounds. In addition, they will become familiar with other NMR active nuclei in conjunction to the proton and carbon spectra.
   - The basic fundamentals of Nuclear Magnetic Resonance (NMR) spectroscopy are covered in lecture and supplemented with supporting information and examples found on blackboard. After covering 1D proton and carbon NMR, 2D NMR experiments will be explored. In classed worked problems are used to illustrate a systemic approach to identifying compound structures and compared to the predicted chemical shifts and coupling patterns to confirm structures. Previous presented techniques, classical analysis and infrared spectroscopy are used in conjunction with NMR to give a more comprehensive identification.
3. Students will be able to use mass spectrometry to determine the chemical formula and interpret electron impact mass spectra for confirmation or identification of compounds. They will also be able to assess different types of mass spectrometry for specific applications used through out the different areas of chemistry.
   - The fundamentals of mass spectrometry are covered in lecture and supplemented with supporting information and examples found on blackboard. The advantages and limitations of the different types of hyphenated mass spectrometers are also covered, highlighting their application to different disciplines of chemistry and the types of molecules studied in those areas. In class problems are worked to give practice and illustrated a systemic approach to identify the molecular formula and functional groups. As before, previous spectroscopic techniques and physical data are used in conjunction with mass spectrometry to give a comprehensive identification of compounds.
4. Students will become proficient in using different spectroscopic techniques to establish the compound purity and evaluate different methods to determine yields as well as percent enantiomer and diastereomer yields and purity.
   - Students will examine and assess the advantages, limitations and accuracy of different

methods in determining purity and yields. Especial emphasis is centered on enantiomers and diastereomers. These topics are covered in lecture and supplemented with materials found on blackboard.

5. Students will be able to critically evaluate and interpret spectral data for the full and complete characterization of organic and organometallic compounds as well as identify unknown compounds. These characterizations will meet the standards for publications in American Chemical Society journals and used through out US patent applications.
   - Students will be able to "read" UV-Vis, Infrared, NMR and Mass Spectroscopic spectral data, being able to extract the critical information, organizing this into an acceptable format for publication in the internationally acceptable ACS journals. Students are expected to write an organized analysis of both spectroscopic and physical data that clearly supports the identification of either prepared compounds or unknowns. This written component is used through out this class and is also used in the oral presentations in class.

- **Real Life Relevance:** This course prepares students for working in industry or academic research involving synthesis or identification of organic compounds. This course is also critical for students doing synthetic work as part of their research project.
- **Relation to Other Courses:** This class is a continuation of material covered in lower division classes such as Chem 232, 457 and 550

Course Materials

- All of the material for this class will be found as PDF documents on the class Blackboard site and in class lecture notes.
- A highly recommended, but optional text, "Organic Structure Analysis" by P. Crews, J. Rodrigues and M. Jaspars is highly recommended. Other useful texts are listed at the end of the syllabus.

Course Conduct

- Attendance and participation in lectures are most strongly recommended in aiding in mastery of spectroscopy, gauge your progress and reinforce fundamentals.
- PDF copies of the PowerPoint slides will be made available via Blackboard shortly before new topics are begin in lecture.
- A total of at least 10-problem set will be distributed during the semester. These will be lightly graded with a maximum value of 10 points. They must be turned by the due date, generally just prior to the start time of lecture. The problem sets must be submitted as a single PDF file that includes your name within the problem set and as part of file name. The image quality must be suitable to be printed and readable, free of background colors and acceptable contrast. Unsuitable files will have a deduction of points and may not be graded due to poor quality. I will not accept last problem sets after the keys are posted on blackboard. Re-grades are accepted within 1 week from the time that keys are posted. Everyone one present one problem in class as a chalk talk to the class. Their grade for that one problem will be 10 points, replacing that problem set. The other half of the problem sets will be evaluated by an individually in class chalk talk. Students will be able to choose which problem to present when they are distributed in class. If additional problem sets are given out, lowest scores on previous problem sets will be replaced with higher scores on additional problem sets. All problem sets answers must reflect the student's own work, (see below).

Exams

Examinations:

Midterm Exam #1	2/25/21	100 pts
Midterm Exam #2	4/1/21	100 pts
Final Exam	TBA	150 pts

Two midterm exams will be given during regular lecture times (February 25 and April 1) from 5:00 PM - 7:00 PM. These exams will cover material present in lecture shortly before the exams. While all exams are accumulative, the emphasis is on material

covered since the previous exams. The final exam date and time will be announced in class after it becomes available. No make up exams will be given during the semester, it is important that you plan your schedule accordingly. The two 2 hour midterm exams will each be worth 100 points. The final is also a 2-hour exam worth 150 points. Half of this exam is focused on material since midterm exam 2 and the other half is on new material. Excused absences, substantiated by an appropriate written confirmation, will result in no penalty. Unexcused absences will result in a “zero” and will account for an “F” grade for such exam. Make-up exams will only be offered in exceptional circumstances, typically requiring advance notice.

Re-grading of exams must be submitted within one week of posting or exam keys or return of exams which ever comes last. Keys for final exams are not posted nor are exams returned to students. However, students may view final grades exams. Math errors on grading have to time limits.

Graduate Student additional assignment

Graduate students will submit a 5 page new application of a spectroscopic technique to their graduate research project. This short proposal will include the basics of this spectroscopic method, and how it relates to their research project and discussion how this application can is advantageous over existing methods. Also describe in this proposal how this method is developed and optimized.

Grading

Your grade will be determined at the end of the semester. Graduate and undergraduate students are graded using separate curves. Graduate student averages are approximately a B+ while undergraduate students average will be about B or slightly higher.

Grading: Your course grade will be based on 450 points maximum. Your grade will be based on your performance from exams and problem sets.

	Undergraduate Students			Graduate Students	
In summary;	10 Problem Sets @10 pts	100 points	22%	100 points	20%
	Midterm Exam 1	100 points	22%	100 points	20%
	Midterm Exam 2	100 points	22%	100 points	20%
	New Spectroscopic Application report			50 points	10%
	Final Exam	150 points	33%	150 points	30%
	Total	450 points	100%	500 points	100%

Students with Disabilities

If you are a student with a disability and believe you will need accommodations for this class, it is your responsibility to contact Student Disability Services at (619) 594-6473. To avoid any delay in the receipt of your accommodations, you should contact Student Disability Services as soon as possible. Please note that accommodations are not retroactive, and that accommodations based upon disability cannot be provided until you have presented your instructor with an accommodation letter from Student Disability Services. Your cooperation is appreciated.

Academic Honesty

The University adheres to a strict policy regarding cheating and plagiarism. These activities will not be tolerated in this class. Become familiar with the policy (http://www.sa.sdsu.edu/srr/conduct1.html). Any cheating or plagiarism will result in failing this class and a disciplinary review by Student Affairs.

Examples of Plagiarism include but are not limited to:

- Using sources verbatim or paraphrasing without giving proper attribution (this can include phrases, sentences, paragraphs and/or pages of work)

- Copying and pasting work from an online or offline source directly and calling it your own
- Using information you find from an online or offline source without giving the author credit
- Replacing words or phrases from another source and inserting your own words or phrases
- Submitting a piece of work you did for one class to another class

If you have questions on what is plagiarism, please consult the policy (http://www.sa.sdsu.edu/srr/conduct1.html) and this helpful guide from the Library:(http://infodome.sdsu.edu/infolit/exploratorium/Standard_5/plagiarism.pdf)

Texts and materials

Recommended Texts

"Organic Structure Analysis" by P. Crews, J. Rodrigues and M. Jaspars

Your class notes will be the basis of the material covered in this class. Fairly extensive spectral data tables will be available on blackboard for your use in this class and hopefully be a value to you afterwards.

Additional Literature References for Qualitative Analysis and Identification of Organic Compounds

Textbooks

Cheronis and Entrikin, *"Identification of Organic Compounds"*
Kamm, *"Qualitative Organic Analysis"*
Elvain, *"The Characterization of Organic Compounds"*
Pasto and Johnson, *"Organic Structure Determination"*
• Schieder, *"Qualitative Organic Microanalysis"*
Shriner, Fuson, Curtin and Morrill, *"The Systematic Identification of Organic Compounds"*
Siggia, *"Instrumental Methods of Organic Functional Group Analysis"*
Wild, *"Characterization of Organic Compounds"*

Reference Sources

Beilstein, *"Handbuch der Organischen Chemie"*, A formula and name index of the volumes is available. There are frequently Beilstein cross-reference numbers available from a variety of sources. The most recent supplement (5th) is in English. This volume is found in the reference room of the Love Library. For a guide on how to use this most important reference work see: Huntress, *"A Brief Introduction to the Use of Beilstein's Handbuch der Organischen Chemie."* It may prove to be most useful.
Feigel, *"Qualitative Analysis by Spot Tests, Volume II, Organic Applications"*
Feiser and Feiser, *"Reagents for Organic Synthesis"*
Frankel and Patai, *"Tables for Identification of Organic Compounds"*
Fitton and Hill, *"Selected Derivatives of Organic Compounds"* (has procedures for preparing derivatives)
Heilbron, *"Dictionary of Organic Compounds"*
Huntress, *"Identification of Pure Organic Compounds"*
"Merck Index"
Mulliken, *"The Identification of Pure Organic Compounds"*
Sandler and Karo, *"Organic Functional Group Preparations"* (comprehensive)
Vecera and Gasparic, *"Detection and Identification of Organic Compounds"* (Techniques for purification (crystallization, distillation, sublimation, extraction, etc., functional group tests, derivative reactions.

Complete literature searches may be made using SciFinder.

Selected References to Spectral Literature

Drago, *"Physical Methods in Chemistry"*
Dyer, *"Applications of Absorption Spectroscopy of Organic Compounds"*
Lambert, Shurvel, Verbit, Cooks, Stout, *"Organic Structural Analysis"*
Pasto and Johnson, *"Organic Structure Determination"*
Sadtler Research Labs, Catalogs of UV, IR and NMR spectra
Williams and Fleming, *"Spectroscopic Methods in Organic Chemistry"*

Infrared

Bellamy, *"The Infra-red Spectra of Complex Molecules"* (one of the most comprehensive books in the field)
Nakanishi, Koji and Solomon, *"Infrared Absorption Spectroscopy"*
Pouchert, *"The Aldrich Library of Infrared Spectra"*
Smith “*Infrared Spectral Interpretation: A Systematic Approach*”

Ultraviolet-Visible

Jaffe and Orchin, *"Theory and Applications of Ultraviolet Spectroscopy"*

Nuclear Magnetic Resonance

Jackman, *"Applications of Nuclear Magnetic Resonance Spectroscopy in Organic Chemistry"*
• Martin and Zektzer, *"Two-Dimensional NMR Methods for Establishing Molecular Connectivity"*
Pople, Schneider and Berstein, *"High Resolution Nuclear Magnetic Resonance"*
Pouchert and Campbell, *"Aldrich Library of NMR Spectra"*

Mass Spectrometry

Biemann, *"Mass Spectroscopy, Organic Chemical Applications"*
Budzikiewicz, Djerassi and Williams, *"Mass Spectrometry of Organic Compounds"*
McLafferty, *"Interpretation of Mass Spectra"*