

# COURSE SYLLABUS

(Tentative\*\*)

CHM 4443

Structural Spectroscopic Methods

4 semester credit-hours

Lect: CFS 104 MWF 9:00-9:50 AM CFS 104

Lab: CFS 313 Wednesday 1:00-4:50 PM CFS 313

(The Student Acknowledgement Form at the end of the syllabus must be completed, signed, and turned in to the Instructor by Jan. 26, 2018)

|                               |   |                  |  |
|-------------------------------|---|------------------|--|
| Instructor:                   | Dr. Benny E. Arney                          | Semester:        | Spring 2018                              |
| Office Phone:                 | 294-1531 off-campus ext.<br>41531 on-campus | Email:           | CHM_BEA@SHSU.EDU                         |
| Office:<br>Check each<br>room | CFS 326<br>Or CFS 305<br>Or CFS 323         | Office<br>Hours: | MW : 9:00-9:30 AM<br>TTh : 8:30-11:00 AM |
| Website                       | Blackboard at<br>www.shsu.edu               |                  |  |

**Textbook (required):** Silverstein, Webster, & Kiemle, "Spectrometric Identification of Organic Compounds", 8th ed., John Wiley & Sons, New Jersey, 2015, ISBN 978-0-470-61637-6.

**Must bring book to every class.**

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## Description:

This course will focus on the elucidation of structural information from a sample using spectroscopic methods such as MS, IR, NMR. The NMR will cover 1 and 2 dimensional techniques as well as heteronuclear observation (i.e. nuclei other than  $^1\text{H}$  or  $^{13}\text{C}$ ). The laboratory will focus on providing a hands-on experience of sample preparation and spectral acquisition. The Primary Focus is deriving the structure of an unknown material by the use of its spectral data alone. Extensive use of problem sets will be made to give the student the practice and experience to decisions.

The laboratory component will consist of becoming proficient in the basic operation of each instrument used including; sample preparation, obtaining routine spectra, interpretation of the spectra obtained, and handling of raw data obtained. Specific Guidelines and Hand-Outs will be provided on BlackBoard and in the laboratory (CFS 313).

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## Prerequisites:

Students in this course must have successfully completed CHM 239 & CHM219 with a grade of C or higher. The students are expected to be proficient in the nomenclature, vocabulary, and principles from General Chemistry and Organic Chemistry which will be included in testing and course materials.

### Study Requirements:

Most of the important learning in this class will be **SELF-LEARNING or SELF-TEACHING**. Class time is best used to work on applying the readings and material. The acquisition of skills comes with the repetition of problem solving. **Attending class and writing notes will NOT be enough for most students to pass the class.** Each student must find the level of interaction with the material necessary to succeed. Study groups may be excellent methods for many students. **Remember you are responsible for your advancement.**

[illegible]

Use of the Internet or reference materials, not specifically designated by the professor for use in this course, to work on problems or assignments will constitute grounds for failure and disciplinary action through the office of the Dean of Students.

### Required Materials:

You are required to bring your book to class. Requisite material from CHM 138/139 includes, but is not limited to, bonding, structure, equilibria, thermodynamics, and nomenclature of common ions, ionic compounds, binary compounds, and acids. Also, you are expected to attempt to work as many of the problems in each chapter as possible.

## Questions in Class:

If you ask a question in class, I generally will not answer it directly, but will ask you questions to find the source of the question or to lead you to the proper question you should have been asking. Most questions arise because of inadequate background or misunderstanding of a prior concept or material. Just providing you an answer to write in your notebook would not solve the problem.

If I ask a question in class, especially one that should be easily answered if a student is keeping up and understanding the material, I will wait for the class to arrive at an acceptable answer. Do not consider this class a spectator class where you show up take notes and then study before the test.

### Attendance Policy:

It will be essential for you to attend class regularly if you desire to perform well. Class attendance will not be used, however, as a criterion for evaluating student performance.

## Writing Standards:

Students enrolled in this course are expected to use literate and effective English in their speech and in their writing. All work submitted must be well-written; grades on written work (including examinations) will be based on expression as well as on content. Remember that improper grammar can drastically modify the meaning of a statement.

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## Objectives: Skills to be Acquired in This Course:

This is a course focused primarily on the interpretation of spectrometric data and its use to deduce as much structural information as possible with regards to the sample available. Regardless of your specific major, the demands of the course will be same for all students as will also the testing. It is an overall goal of this course to improve the problem solving skills and understanding of chemical systems of all students enrolled.

1. General understanding of the correspondence between molecular structure and spectroscopic response.
2. Specific information available from each spectrometric method.
3. Information not available from each spectrometric method.
4. Ability to discern structural data and connect them together.

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## Examinations & Quizzes:

The grading for this course will be composed of three components:

|                                      |             |
|--------------------------------------|-------------|
| A. Problem Sets (5@)                 | 40 %        |
| B. Lab Assignments (5@)              | 30 %        |
| <u>C. Mid-Term&amp;Final (15% @)</u> | <u>30 %</u> |
| Total                                | 100 %       |

**Problem Sets:** There will be two types of problem sets those that we work as a class to illustrate the approaches one can take to solving these problems and those assigned to be worked for a grade. Those assigned for a grade must be worked independently without any assistance, aside from the specific texts or materials allowed by the instructor. The students may not discuss them with each other with the lone exception of asking specific questions to the instructor during the assigned class time so that all may hear both the question and any answer that the instructor may provide.

Each lab assignment will be composed of 2 parts, the first of which is usually performable within a single lab period but some of which may require an extensive amount of tinkering to “get it just right”. The second part will be obtaining presentable spectra of a set of unknown compounds that will be used to perform their identification. The last lab assignment will be the identification of a group of unknowns.

The mid-Term and Final will be two (2) 2-hour Blue-book tests. The spectral interpretation problems for the Midterm and Final will be made available on Blackboard two (2) days prior to the inclass test to allow you time to go through the problems and derive the information requested. However, during the in class portion, it is the derivation of the information that is of importance for grading, so no outside work may be brought in for the test. The Midterm will be taken at the syllabus assigned time in the laboratory time slot and the Final will taken at the University assigned time in the class-room space.

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## Test Format & Content:

Each test will be comprehensive and open-book. However, it must be remembered that the book will only be useful if you have been continuously using it for problems. Working the

problems at the ends of each chapter is the best way to study and evaluate your progress in this course and prepare for the tests.

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## Make-up Tests:

There are none. Do not ask for them.

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## Late Work:

Will not be accepted and will be recorded as zeros.

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## Grading:

If a student earns  $\geq 40\%$  on the final, a letter grade will be assigned based on their total accumulated points:

| Grade | Undergraduate | Graduate   |
|-------|---------------|------------|
| 4 (A) | 90 - above    | 93- above  |
| 3 (B) | 80 – 89.99    | 84 – 92.99 |
| 2 (C) | 70 – 79.99    | 75 – 83.99 |
| 1 (D) | 60 – 69.99    |            |
| 0 (F) | <60.00        | <75.00     |

If a grade of less than 40% is earned on the final, an F will be awarded regardless of previous points.

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## ADA Policy:

SHSU adheres to all applicable federal, state, and local laws, regulations, and guidelines with respect to providing reasonable accommodations for students with disabilities. If you have a disability that may affect adversely your work in this class, then I encourage you to register with the SHSU Counseling Center and to talk with me about how I can best help you. All disclosures of disabilities will be kept strictly confidential. NOTE: no accommodation can be made until you register with the Counseling Center.

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## Academic Dishonesty (Cheating) Policy:

*“All students are expected to engage in all academic pursuits in a manner that is above reproach. Students are expected to maintain complete honesty and integrity in academic experiences both in and out of the classroom. Any student found guilty of dishonesty in any phase of academic work will be subject to disciplinary action. The University and its official representatives may initiate disciplinary proceedings against a student accused of any form of academic dishonesty including, but not limited to, cheating on an examination or other academic work which is to be submitted, plagiarism, collusion and the abuse of resource materials.”* And for this class, use of any resource other than those specifically designated by the instructor (especially the internet) will be grounds for automatic failure and possible disciplinary action from the Dean of Students.

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## Inappropriate Classroom Conduct Policy:

*“Students will refrain from behavior in the classroom that intentionally or unintentionally disrupts the learning process and, thus, impedes the mission of the university. Cellular telephones and pagers must be turned off before class begins. Students are prohibited from eating in class, using tobacco products, making offensive remarks, reading newspapers, sleeping, talking at inappropriate times, wearing inappropriate clothing, or engaging in any form of distraction. Inappropriate behavior in the classroom shall result in a directive to leave class. Students who are especially disruptive also may be reported to the Dean of Students for disciplinary action in accordance with university policy.”*

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## Visitors to the Classroom:

*“Unannounced visitors to the class must present a current, official SHSU identification card to be admitted in the classroom. They must not present a disruption to the class by their attendance. If the visitor is not a registered student, it is at the instructor’s discretion whether or not the visitor will be allowed to remain in the room.”*

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## Schedule for Lecture Topic: CHM443

| Date   | Topic  | Reading Assignment |
|--------|--|--------------------|
| Jan 17 | Introduction   | Chap 1             |
| 19     | Mass spectrometry: <b>Ionization Methods</b>   |                    |
| 22     | <b>Interpretation of EI and CI MS:</b> Molecular formula, Isotopic distributions, HRMS, Beynon Tables, Degrees of Unsaturation.  |                    |
| 24     | <b>Fragmentation and Rearrangements:</b> General, hydrocarbons, alkyl halides.   |                    |
| 26     | Alcohols, ethers, ketones, aldehydes, carboxylic acids and derivatives   |                    |
| 29     | Nitrogen containing compounds: amines, amides, nitro compounds, nitriles, nitrite, and nitrates.   |                    |
| 31     | Problems and questions: various MS-structural problems<br><a href="#">Twelfth Class Day. Last day to drop without a "Q" and receive 100% refund. (This does not apply to dropping your only course. Please refer to the <a href="#">Schedule of Classes</a> for refund information.)</a> |                    |
| Feb 2  | <b>Infrared:</b> Introduction, theory, and hydrocarbons.   | Chap 2             |
| 5      | Hydrocarbons, alcohols, ethers, ketones, and aldehydes   |                    |
| 7      | Carboxylic acids and derivatives   |                    |
| 9      | Amines, amides, nitriles, amine-salts, <b>Problem Set #1 DUE</b>   |                    |
| 12     | Nitro, nitroso, cumulated compounds  |                    |
| 14     | Problems and questions: various IR/MS structural problems.   |                    |
| 16     | <b><sup>1</sup>H Nuclear magnetic resonance:</b> Introduction & theory, spin, magnetic fields, excitation, and detection.  | Chap 3             |
| 19     | Chemical Shift Equivalency, Chemical shifts, spin-coupling   |                    |
| 21     | Spin systems, exchangeable protons, magnetic equivalency.  |                    |

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|-------|---|--------|
| 23    | Coupling to other Nuclei: $^2\text{H}$ , $^{19}\text{F}$ , $^{31}\text{P}$ , $^{29}\text{Si}$ , and $^{13}\text{C}$   |        |
| 26    | Chirality, vicinal and geminal coupling, Decoupling-Double Resonance, <b>Problem Set#2 DUE</b>  |        |
| 28    | NOE-spatial proximity   |        |
| Mar 2 | Problems and questions: Various $^1\text{H}$ /IR/MS structural problems   |        |
| 5     | <b><math>^{13}\text{C}</math> Nuclear magnetic resonance:</b> theory, $T_1$ relaxation, NOE, $^{13}\text{C}$ - $^1\text{H}$ coupling, sensitivity, Broadband Decoupled. | Chap 4 |
| 7     | Off-resonance decoupled, gated decoupled (quantitative), gated coupled and DEPT,  |        |
| 9     | Chemical shifts, Chemical shift equivalency   |        |
| 19    | Chemical shifts of major classes  |        |
| 21    | Empirical calculation of $^{13}\text{C}$ Chemical Shifts, <b><u>MID-TERM (March 12) Problem Set#3 DUE</u></b>   |        |
| 23    | Problems and questions: Various $^{13}\text{C}$ / $^1\text{H}$ /IR/MS structural problems   |        |
| 26    | <b>Correlation NMR :</b> theory   | Chap 5 |
| 28    | $^1\text{H}$ - $^1\text{H}$ correlation: COSY & $^{13}\text{C}$ - $^1\text{H}$ COSY: HECTCOR  |        |
| Apr 2 | Double Quantum Filtered, Inverse-detection Methods, HMQC, HMBC.   |        |
| 4     | $^{13}\text{C}$ - $^{13}\text{C}$ COSY: Inadequate, TOCSY, ROESY(NOESY) <b>Problem Set#4 DUE</b>  |        |
| 6     |   |        |
| 9     | Problems and questions  |        |
| 11    | <b>NMR of Other important Spin <math>\frac{1}{2}</math> Nuclei:</b>   | Chap 6 |
| 13    | $^{15}\text{N}$ , $^{19}\text{F}$   |        |
| 16    | $^{29}\text{Si}$ , $^{31}\text{P}$  |        |
| 18    | <b>Problems and questions:</b>  |        |
| 20    | Solved Problems and problems from other sources.  | Chap 7 |
| 23    | Problems and questions  |        |
| 25    | Problems and questions  |        |
| 27    | Problems and questions  |        |
| 30    | Problems and questions  |        |
| May 2 | Problems and questions <b>Problem Set#5 DUE</b>   |        |
| 4     | Final Exam Study Day. Last Day to Resign (SEE <b>RESIGNATION INFORMATION BELOW</b> ).Last day for dropping Spring Semester courses without grade of F                   |        |
| 7-10  | <b>!!!!!! FINAL EXAMINATIONS !!!!!!!</b>  |        |
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**\*\* This document is tentative in that changes may be made as deemed necessary by the Professor in order to achieve the objectives of the course.**

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## Student Acknowledgement of Syllabus:

I, \_\_\_\_\_ (*your name*) having SHSU ID# \_\_\_\_\_, have printed the syllabus for CHM 443 (Spring 2007). I further acknowledge that I have read the syllabus and that I am familiar with and understand its contents. I also recognize that my continuance in this course requires that I agree to its content and requirements and that changes to this syllabus are only possible if they further the aims of the course as deemed appropriate by the professor.

I am also aware that questions and/or problems with the course must be addressed to the instructor. If these problems are not part of the day's scheduled material, it should be addressed after class, during office hours, or by appointment.

Signed : \_\_\_\_\_

Date: \_\_\_\_\_