SOUTHERN CONNECTICUT STATE UNIVERSITY

CHE 440  Instrumental Methods of Analysis
Spring Semester 2010
Tuesday, Thursday 9:35 – 10:50 am (lecture)
Tuesday 2:00 – 6:00 pm (laboratory)

Name: Dr. Robert J Snyder  
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Office Hours:
Tuesday, Wednesday, Thursday 11-12 p.m.
Wednesday 1-3 p.m.
or at other times by appointment

COURSE NUMBER    CHE 440  CREDIT HOURS    4  PREREQUISITES:
CHE 240, 260, 261

COURSE TITLE: Instrumental Methods of Analysis

COURSE DESCRIPTION:

<table>
<thead>
<tr>
<th>Expected Student Learning Activity</th>
<th>Weekly Hours for Course</th>
<th>Total Hours for Course (14 week semester)</th>
<th>Term Credits Earned</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lecture Time (Contact Hours)</td>
<td>3</td>
<td>42</td>
<td></td>
</tr>
<tr>
<td>Reading and Study Time</td>
<td>8</td>
<td>112</td>
<td></td>
</tr>
<tr>
<td>Laboratory (Contact Hours)</td>
<td>4</td>
<td>56</td>
<td></td>
</tr>
<tr>
<td>Laboratory Reports</td>
<td>7</td>
<td>98</td>
<td></td>
</tr>
<tr>
<td>Examinations (2 hr during lecture)</td>
<td></td>
<td>4 (mid-term + final)</td>
<td></td>
</tr>
<tr>
<td><strong>Total Hours</strong></td>
<td><strong>22</strong></td>
<td><strong>312</strong></td>
<td><strong>4</strong></td>
</tr>
</tbody>
</table>

Chemistry 440, Instrumental Methods of Analysis, is an introduction to the theoretical background and practical use of modern instruments in the analytical laboratory. After a brief introduction to the use of instrumentation for chemical analysis, the properties of electromagnetic radiation will be discussed. Spectroscopic techniques that will be covered include: Ultraviolet/Visible Spectrophotometry, Fluorescence and Phosphorescence, Atomic Absorption (Flame and Graphite Furnace) Spectroscopy, Infrared Spectroscopy and Nuclear Magnetic Resonance Spectroscopy. After an introduction to chromatographic separations, Gas Chromatography and High-Performance Liquid Chromatography will be discussed.

Students will conduct experiments during the laboratory portion of the course that will demonstrate the scientific method and illustrate basic concepts presented in the lecture portion of the course. The experiments chosen will demonstrate the practical use of modern instrumentation in solving real-world problems, such as determining the amount of lead in Southern’s drinking water by Graphite Furnace Atomic Absorption Spectrometry; the amount of calcium, iron and copper in Total K breakfast cereal by Flame Atomic Absorption Spectrometry; the amount of aspirin and caffeine in Anacin tablets by Nuclear Magnetic Resonance Spectroscopy; and the amount of caffeine in cola drinks by High-Performance Liquid Chromatography.
CHE 440 is a junior/senior level course in Analytical Chemistry. It is generally considered to be the second half of a year of Analytical Chemistry that began with CHE 240, Quantitative Analysis. This course is a requirement for students pursuing the science education degree in chemistry. The lecture portion of the course concentrates on the theoretical aspects of the modern instrumentation that today’s chemists are using in the analytical laboratory. The laboratory portion emphasizes the practical use of those instruments that the American Chemical Society (ACS, chemistry’s external accrediting agency) has designated as required for an ACS-certified degree in chemistry.

The course emphasizes analytical thinking and problem solving using both quantitative and qualitative methods of analysis. Students are expected to learn how to operate the various spectroscopic and chromatographic instruments as well as learn the best way to prepare their samples for analysis.

The laboratory experiments offer the students an inquiry-based approach to solving problems. The instructor provides the students with detailed instructions on the operation of the instruments, but he students are left on their own as to designing the experiment. The students also learn proper laboratory conduct, good laboratory practice, concern for the environment and instructions on safety in science.

**LEARNER OUTCOMES AND ASSESSMENT**

1. Understand the methods and techniques of Analytical Chemistry. Students will be expected to differentiate among the various areas of Chemistry and explain specifically what an Analytical Chemist does. (INTASC 1; NSTA 1, 3, 4; CCCT 1, 4, 2.6)

2. Differentiate between qualitative analysis and quantitative analysis and learn which instrumental techniques are bested suited for each task. Students will perform laboratory experiments using certain instruments to identify unknowns (qualitative) and other instruments to determine the composition of the unknown (quantitative). (INTASC 1, 4; NSTA 1, 3, 4; CCCT 1.1, 1.3, 1.4, 2.6)

3. Learn the difference between classical methods of analysis and instrumental methods of analysis. Students will be able to identify and explain the purpose of the classical wet techniques and the modern instrumental methods of analysis. (INTASC 1, 4; NSTA 1, 2, 3; CCCT 1.1, 1.3, 1.5, 2.6)

4. Define analyte and analytical signal. Students will be able to identify what each instrument is actually measuring and how it is determining the measurement. (INTASC 1, 4; NSTA 1, 2, 3; CCCT 1.1, 1.3, 1.4, 2.6)

5. Identify the four basic components of any instrument. For every instrumental technique that the student uses, they will be expected to identify the 4 basic components and their functions. (INTASC 1, 4; NSTA 1, 2, 3; CCCT 1.1, 1.3, 1.4, 2.6)

6. Evaluate instrumental techniques by determining the six performance characteristics of analytical instruments. Students will be expected to explain and calculate, in a formal written laboratory report, how each instrument performs in the six categories. (INSTAC 1, 4; NSTA 1, 2, 3, 4; CCCT 1.4, 1.6)

7. Use the method of least squares (linear regression) to determine calibration curves for quantitative analysis. When using an instrument for quantitative analysis, students will be expected to plot the data obtained in such a manner as to get a linear relation between the analytical signal and the concentration of the analyte. (INTASC 1, 4; NSTA 1, 2, 3, 4; CCCT 1.4, 1.6)

8. Understand the difference between signals and noise. Students will be expected, for each instrument studied, to be able to calculate the signal to noise ratio from experimental data. (INTASC 1, 4; NSTA 1, 2, 3, 7; CCCT 1.4)
9. Learn the difference between noise reduction and signal enhancement. Students will perform laboratory experiments in which they will learn the two ways to increase the signal to noise ratio of each instrument studied. (INTASC 1; NSTA 1, 2, 3; CCCT 1.2, 1.3, 1.4, 2.2, 2.6)

10. Identify chemical noise, instrumental noise, and environmental noise. Students will be expected to write short essays on examination to explain the origin of the various kinds of noise that arises in the instruments studied. (INTASC 1.2, 1.3, 1.4, 1.6, 2.1, 2.6)

11. Differentiate between hardware devices to reduce noise and software methods to enhance signals. Students will be expected to explain how different instruments reduce the amount of noise or increase the strength of the analytical signal to achieve the increase in signal to noise ratio desired. (INSTAC 1.4; NSTA 1, 2, 3, 4, 7; CCCT 1.2, 1.4, 2.2, 2.6)

12. Identify the various regions of the electromagnetic spectrum and what information instrumental techniques tell us about each region. Students will perform laboratory experiments investigating how different sources of radiation yield different kinds of information about the analyte. (INSTAC 1, 4; NSTA 1, 2, 3, 4; CCCT 1.2, 1.3, 1.4, 1.6, 2.2)

13. Discuss the wave-particle duality of matter and energy. Students will learn in lecture and be asked on examinations, to explain the significance of the dual interpretation of matter and energy on this planet. (INSTAC 1, 4; NSTA 1, 2, 3; CCCT 1.3, 1.4)

14. Calculate wavelength, frequency and wavenumber for the different regions of the electromagnetic spectrum. Students will be expected to do homework problems to convert from one parameter to the other, used in describing the different ways in which energy is used in instrumental analysis. (INSTAC 1, 4; NSTA 1, 2, 3; CCCT 1.2, 1.4)

15. Differentiate among diffraction, refraction, reflection and scattering of radiation. In addition to the interactions of matter and energy that lead to spectroscopic techniques, (INSTAC 1, 4; NSTA 1, 2, 3; CCCT 1.2, 1.3, 1.4, 2.2)

16. Use the Planck equation to calculate energy for the various regions of the electromagnetic spectrum. (INSTAC 1, 4; NSTA 1.2, 3; CCCT 1.4)

17. Discuss the impact of the Heisenberg Uncertainty principle on spectroscopy. (INSTAC 1, 4; NSTA 1, 2, 3; CCCT 1.2, 1.4, 2.2)

18. Learn the Born-Oppenheimer approximation and why it makes spectroscopy possible. (INSTAC 1, 4; NSTA 1, 2, 3, 7; CCCT 1.4)

19. Understand the various processes (radiative and nonradiative) by which atoms or molecules emit energy upon absorption of radiation. (INSTAC 1, 4; NSTA 1, 2, 3; CCCT 1.3, 1.4)

20. Identify the various components (radiation sources, wavelength selectors, entrance and exit slits, sample containers, radiation detectors) of optical instruments. (INSTAC 1, 4; NSTA 1, 2, 3, 4; CCCT 1.3, 1.4, 2.2)

21. Use ultraviolet/visible spectroscopy for quantitative analysis. (INSTAC 1, 4; NSTA 1, 2, 3, 4; CCCT 1.4)

22. Identify the various forms of atomic spectroscopy and their uses. (INSTAC 1, 4; NSTA 1, 2, 3, 4, 7; CCCT 1.2, 1.4, 1.6, 2.2)

23. Calculate the sensitivity (characteristic concentration) of a Flame Atomic Absorption
Spectrophotometer and the sensitivity (characteristic mass) of a Graphite Furnace instrument. 
(INSTAC 1 ; NSTA 1, 3, 4 ; CCCT 1.4, 2.6)

24. Understand what is happening to the sample in the graphite tube during each of the five steps of a Graphite Furnace method. (INSTAC 1, 4 ; NSTA 1, 3, 4 ; CCCT 1.1, 1.3, 1.4, 2.6)

25. Identify the various components (nebulizers, atomizers, radiation sources, monochromators, radiation detectors) of flame atomic absorption and graphite furnace atomic absorption instruments. (INSTAC 1, 4 ; NSTA 1, 2, 3, 4 ; CCCT 1.1, 1.3, 1.4, 2.6)

26. Identify the infrared region of the electromagnetic spectrum and understand what information is obtained from it. (INSTAC 1, 4 ; NSTA 1, 2, 3 ; CCCT 1.1, 1.3, 2.6)

27. Connect the theoretical ideas of the classical model and quantum mechanical model of infrared spectroscopy with molecular structure. (INSTAC 1, 4 ; NSTA 1, 2, 3, 4, 5, 7 ; CCCT 1.3, 1.5, 2.6)

28. List the two necessary conditions for infrared absorbance. (INSTAC 1, 4 ; NSTA 1, 2, 3 ; CCCT 1.1, 1.3, 2.6)

29. Predict which infrared sample handling technique is best suited for the analysis. (INSTAC 1, 4 ; NSTA 1, 2, 3, 4, 7 ; CCCT 1.1, 1.3, 1.5, 2.6)

30. Describe a modern Fourier-Transform Infrared Spectrometer, including the interferometer, and how it collects data. (INSTAC 1, 4 ; NSTA 1, 2, 3, ; CCCT 1.1, 1.3, 2.6)

31. Recognize how nuclear magnetic resonance spectroscopy differs from the other spectroscopic techniques. (INSTAC 1, 4 ; NSTA 1, 2, 3, 4, 7 ; CCCT 1.1, 1.3, 1.4, 2.6)

32. Calculate the resonance frequency, the number of magnetic energy states and the magnetic quantum number for each state of a particular isotope. (INSTAC 1, 4 ; NSTA 1, 2, 3, ; CCCT 1.1, 1.3, 1.4, 2.6)

33. Describe the function of each component of a Nuclear Magnetic Resonance Spectrometer. (INSTAC 1, 4 ; NSTA 1, 2, 3, 4 ; CCCT 1.1, 1.3, 1.4, 2.2)

34. Use chemical shifts and multiplets from spin-spin splitting to predict molecular structure of compounds from Nuclear Magnetic Resonance spectra. (INSTAC 1, 4 ; NSTA 1, 2, 3 ; CCCT 1.2, 1.4, 1.6, 2.2)

35. Classify the various chromatographic techniques by their mobile phases and mode of separation. (INSTAC 1, 4 ; NSTA 1, 2, 3, 4 ; CCCT 1.2, 1.4, 1.5, 2.5, 2.6)

36. Understand the difference between gas chromatography and liquid chromatography and what kind of samples are best analyzed by each technique. (INSTAC 1, 4 ; NSTA 1, 2, 3, 4 ; CCCT 1.2, 1.3, 1.4, 1.6, 2.2, 2.6)

37. Calculate linear velocity of the mobile phase, the number of theoretical plates, the plate height and the resolution for a chromatographic column. (INSTAC 1, 4 ; NSTA 1, 2, 3, 4 ; CCCT 1.2, 1.3, 1.4, 1.6, 2.2, 2.6)

38. Interpret van Deemter plots and how they illustrate column processes and band broadening. (INSTAC 1, 4 ; NSTA 1, 2, 3, 4 ; CCCT 1.2, 1.3, 1.4, 2.2, 2.3, 2.6)
**MODES OF LEARNING:**
Class lecture and discussion; problem solving; analytical troubleshooting; experiment design; acquisition and both quantitative and qualitative interpretation of spectroscopic and chromatographic data.

**COURSE CONTENT OUTLINE:**

<table>
<thead>
<tr>
<th>CHAPTER</th>
<th>TOPIC</th>
<th>NUMBER OF LECTURES</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>INTRODUCTION</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>Introduction to instrumental methods, classification of instrumental techniques, basic functions of instrumentation, performance characteristics of instruments</td>
<td>3.5 lectures</td>
</tr>
<tr>
<td>5</td>
<td>Signals and noise, sources of noise in Instrumental Analysis, Signal to Noise enhancement, software methods for signal to noise enhancement</td>
<td>1.5 lectures</td>
</tr>
<tr>
<td></td>
<td><strong>SPECTROSCOPIC TECHNIQUES</strong></td>
<td></td>
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<tr>
<td>6</td>
<td>Properties of electromagnetic radiation, the wave-particle duality of nature, absorption and emission of radiation</td>
<td>2.75 lectures</td>
</tr>
<tr>
<td>7</td>
<td>Components of instruments for Optical Spectroscopy</td>
<td>3 lectures</td>
</tr>
<tr>
<td>13</td>
<td>An introduction to Molecular ultraviolet/visible and near-infrared absorption spectroscopy, fundamental laws of photometry</td>
<td>1 lecture</td>
</tr>
<tr>
<td>14</td>
<td>Applications of ultraviolet/visible molecular absorption spectrometry, single component analysis, multicomponent analysis, method of standard additions</td>
<td>1.25 lectures</td>
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<td></td>
<td><strong>MIDTERM EXAMINATION</strong></td>
<td></td>
</tr>
<tr>
<td>16 and 17</td>
<td>Infrared Absorption Spectroscopy, theory, models, sample handling techniques, instrumentation, classical dispersive and modern Fourier-Transform</td>
<td>5.25 lectures</td>
</tr>
<tr>
<td>8 and 9</td>
<td>Atomic Spectroscopy based upon Flame and Electrothermal Atomization, atomic line widths, atomization, nebulization, sensitivity, interferences, components of instruments.</td>
<td>4.25 lectures</td>
</tr>
</tbody>
</table>
interpretation of infrared spectra

19 Nuclear Magnetic Resonance Spectroscopy, theory, relaxation, continuous wave and pulsed (Fourier-Transform) instruments, NMR spectra and molecular structure, the chemical shift, spin spin splitting, identification of compounds by NMR spectroscopy 3.75 lectures

CHROMATOGRAPHIC TECHNIQUES

26 Introduction to Chromatographic techniques, classification of chromatographic methods, qualitative and quantitative aspects of chromatography, plate theory and rate theory, the van Deemter equations 2.25 lectures

27 Gas Chromatography If time allows

28 High Performance Liquid Chromatography If time allows

FINAL EXAMINATION

REQUIRED TEXT:


COURSE REQUIREMENTS:

Students will be expected to read the assigned chapters in the textbook prior to classroom lecture and to attempt the suggested problems for each chapter listed on the syllabus. The suggested problems are found at the end of each chapter in the textbook and are representative of the material in that chapter that the professor believes is most important for the student to understand. It of course follows that these are also the types of questions and problems that will appear on the midterm and final examinations.

There will be two examinations in the course; a midterm and a final examination. The examination questions will be taken directly from the material covered in class. There will be both questions requiring short answers or descriptions of instrumental techniques and quantitative problems similar to the suggested problems in the textbook. Both exams will be two hours long; the midterm will cover the first half of the course and the final examination will cover only material from the midterm to the end of the course.

Students are required to complete all laboratory experiments. The students normally work in teams in the laboratory but the laboratory reports must be done by the individual students alone. The reports are due two weeks after completion of the laboratory experiment. It is the policy of the Chemistry Department at Southern Connecticut State University that, to receive a passing grade in any chemistry course with a laboratory component, the student must pass the laboratory section of the course. A passing grade for the laboratory portion of CHE 440 is sixty (60%) percent.
EVALUATION CRITERIA:

- Mid-Term Examination 30%
- Final Examination 30%
- Laboratory Reports 40%
- Total 100%

The actual letter grade will be based on the grading scale listed below with the possible adjustment for class average at the end of the semester (if necessary).

- >95 % A+
- 87 – 94 % A
- 82 – 86 % A-
- 75 – 81 % B+
- 70 – 74 % B
- 66 – 69 % C+
- 61 – 65 % C
- 58 – 60 % C-
- 55 – 57 % D+
- 52 – 54 % D
- 50 – 51 % D-
- <50 % F
<table>
<thead>
<tr>
<th>Scholarship</th>
<th>Professional Standards</th>
<th>CCCT [CONNECTICUT COMMON CORE OF TEACHING]</th>
</tr>
</thead>
</table>
| 1. Knowledge of subject matter  
2. Knowledge of human development & learning  
3. Instruction adapted to meet diverse learners  
4. Use of multiple instructional strategies & resources | National Science Teacher's Association  
1. Content - Structure and interpret the concepts, ideas and relationships in science  
2. Nature of Science - Define the values, beliefs and assumptions inherent to the creation of scientific knowledge within the scientific community  
3. Inquiry - Formulating solvable problems, constructing knowledge from data, exchanging information for seeking solutions, developing relationships from empirical data  
4. Context of Science - Relate science to daily life: technological, personal, social and cultural values.  
5. Skills of Teaching - Science teaching actions, strategies and methodologies, interaction with students, effective organization and use of technology.  
6. Curriculum - Extended framework of goals, plans, materials and resources for instruction.  
7. Social Context - Social | DEMONSTRATIONS OF KNOWLEDGE  
1.1 understanding of student learning & development  
1.2 understanding of need for different learning approaches  
1.3 proficiency in reading, writing and mathematics  
1.4 understanding of central concepts & skills, tools of inquiry and structures of discipline(s)  
1.5 knowledge of how to design and deliver instruction  
1.6 recognition of need to vary instructional methods | APPLICATION OF KNOWLEDGE THROUGH  
2.1 instructional planning based upon knowledge of subject, students, curriculum & community  
2.2 selection and/or creation of learning tasks that make subject meaningful for students  
2.3 establishment and maintenance of appropriate behavior standards and creation of positive learning environment  
2.4 creation of instructional opportunities supporting students' academic, social and personal development  
2.5 use of verbal, nonverbal and media communication fostering individual and collaborative inquiry  
2.6 employment of various instructional strategies in support of critical thinking, |
| 10. Partnership with school and community | and community support network, relationship of science to needs and values of the community, involvement of people in the teaching of science. |
| 8. Assessment | Alignment of goals, instruction and outcomes, evaluation of student learning. |
| 10. Professional Practice | Knowledge and participation in the professional community, ethical behavior, high quality of science instruction, working with new colleagues as they enter the profession. |
| | problem solving and skills demonstration |
| 2.7 use of various assessment techniques to evaluate student learning & modify instruction |

**DEMONSTRATION OF PROFESSIONAL RESPONSIBILITY THROUGH:**

- **3.1** professional conduct in accordance with the Code of Professional Responsibilities for Teachers
- **3.2** shared responsibility for student achievement and well-being
- **3.3** continuous self-evaluation regarding choices & actions on students and school community
- **3.4** commitment to professional growth
- **3.5** leadership in the school community
- **3.6** demonstrations of a commitment to students and a passion for improving the profession

**DISABILITY ACCOMMODATION STATEMENT**

If any student has a particular disability-related need in order to participate in this course, such as, special seating, note-taking assistance, use of tape-recorders, or modified examination conditions, please let me know as soon as possible so that appropriate accommodations can be made. The student will need to make an appointment with the Disability Resource Center located in EN B 222 to arrange for approved accommodations. However, if you have other information you wish to speak to me about, if you have emergency medical information to share with me, or if you need special arrangements in case the building must be evacuated, please make an appointment with me as soon as possible. My office is located in Jennings Hall (JE 305) and my office hours are listed on the first page. Every effort will be made to accommodate students in this course.

**ADDITIONAL COMMENTS**

**Missed/Late Work:**

The formal written laboratory reports are due two (2) weeks after the experiment has been completed. Late laboratory reports will have five (5) points taken off for each day late, the first time. The second time a laboratory
Report is late, ten (10) points a day will be taken off, etc. Remember that, it is SCSU’s Chemistry Department policy that the student must pass the laboratory portion of a course, to pass the course.

Make-up examinations will only be given in the case of substantiated illness (a doctor’s note is required). In this case, the student must inform the professor of his/her illness BEFORE the exam.

Inclement Weather:
When inclement weather threatens, call the University’s WeatherChek voice mail system at 203-392-SNOW to hear the latest official information on possible delayed openings, class cancellations or the closing of the University. If a scheduled examination is postponed due to inclement weather (or for any reason), that examination will be given the next time that the class meets. If a laboratory session is cancelled for any reason, it will be made-up the following Tuesday.

Attendance:
Regular and prompt attendance of scheduled classes and laboratory sessions is necessary for the student to derive the intended benefit of the learning experience the college strives to provide and for the optimization of the student’s academic progress. This especially impacts itself in a course like chemistry, where every day’s lecture requires that the student has given consideration and attempted to come to grips with the material discussed in the preceding class session.

Cell Phones:
All cell phones and pagers must be turned off during the lecture. Students who ignore this policy will be asked to leave the classroom. If you are on call for work related emergencies or personal reasons, please switch to a mode that will not disturb the class (i.e. vibrate mode) and inform the professor prior to class.

Academic Dishonesty:
The student is challenged to put forth a maximum effort while preparing for this class. As with any worthwhile endeavor, the more the student puts in, the more that they will get out, i.e., learn from the actual experience. The professor will, concurrently, be putting forth a maximum effort to bridge any gaps between preparation and performance, so that the student’s maturity, creativity and curiosity as students of Analytical Chemistry may be developed, encouraged and stimulated.

In this vain, cheating in any form will not be tolerated. Academic dishonesty is defined in the SCSU Student Handbook and other University publications and includes either giving or receiving information on homework assignments, mid-term or final examinations and laboratory reports. As a minimum, a grade of zero will be given as a result of cheating on any one assignment. Continued evidence of academic dishonesty may result in expulsion from the course.

The student’s cooperation and consideration of these guidelines is essential for maximum fruition of the learning experience.