Syllabus CHEM 4421
Instrumental Analysis - Spring 2020

Instructor
Dr. David Carter
Office hours: M 10:30-11:30 AM, W 8:00-11:00 AM, R 11:30-12:00 AM or by appointment
Office: CAV 218     Office Phone: 486-6626
email: david.carter@angelo.edu  Course web site: http://blackboard.angelo.edu

Time and Location
Class times: TuTh 8:00-9:15 A.M.  CAV 223
Lab: Th 2:00-4:50 P.M., CAV 206/203   (Prelab: CAV 211)

Books and Supplies
Texts: Quantitative Chemical Analysis, 8th ed., Daniel C. Harris, 2010 (Sapling eBook recommended)
Online Homework:  Sapling Learning (Angelo State University - CHEM 4421 - Spring15 - CARTER)
Other Materials: The Official Laboratory Research Notebook and Safety Goggles

Purpose of Course
Instrumental analysis is the cornerstone of the majority of work performed in the physical and life sciences. From determining the composition of soil on Mars to the changes of neurotransmitters in single nerve cells, from determining the ages of rocks in billions of years to monitoring the progress of a catalytic reaction on the femtosecond timescale, you will find instrumental methods in the center of the action. This course is designed to open the door of understanding of instrumental methods to you. The list of instrumental methods may seem endless, the complexity of the machines mind-boggling and the wealth of information produced overwhelming. HOWEVER, instrumental methods are designed around an amazingly small number of basic principles so that with a reasonable effort you can have insight into every area of scientific endeavor. An understanding of instrumental methods will help you to understand how your auto exhaust is measured or the inner-workings of a medical technology lab. A good understanding of instrumental methods will prepare you for graduate school or medical school and is one of the best skills that you can have if you are seeking employment as a B.S. chemist. This course should revolutionize how you see the world.

Evaluation of Student Learning Outcomes
Student learning outcomes will be evaluated by test questions and by the grading of lab notebook, reports and other assignments.

Grading
Classroom participation, Quizzes, Non-Sapling Homework  250 pts  A . . . ≥880
Sapling Homework (165 pts possible)  150 pts  B . . . 760-880
Exams  100 pts  C . . . 650-760
Lab (325 pts possible)  300 pts  D . . . 550-650
Final Exam  200 pts  F . . . <550
Total  1000 pts
### Student Learning Outcomes

**Essential Learning Outcomes**

Number-letter notations in parentheses refer to Chemistry Department Student Learning Objectives which are available at the [Chemistry Department website](#).

**Learning to apply course material**

(to improve thinking, problem solving, and decisions)

- Students coming out of the course should be able to decide what technique should be used for a critical application. They should be able to justify their choice and identify crucial method conditions for the application.
- Students should be able to apply the general principles of instrumental methods to understand and evaluate unfamiliar or new instrumental techniques.

**Developing specific skills and competencies**

(needed by professionals in the fields most closely related to this course)

- Students should know key skills needed to produce reliable data and make good decisions regarding that data. This requires the ability to do the following
  - Optimize instrument performance
  - Process and analyze data
  - Meaningfully Interpret Data (requires awareness of method limitations)
  - Understand at a technical level how instruments work

**Important Goals**

**Gaining factual knowledge**

(terminology, classifications, methods, trends)

- Students will be able to identify and name the major instrumental methods.
- Students will be able to classify methods according to the following criteria:
  - Type of method (spectroscopic, chromatographic, electrochemical, etc.)
  - Type of analyte
  - Information required
  - Concentration range (major component, minor component, trace, etc.)

**Learning fundamental principles, generalizations, or theories**

- Students will be able to describe the basic principles involved in the major instrumental methods and explain how these principles enable a method to obtain the desired information.
- Students will be able to used quantitative figures of merit to evaluate the suitability of a method for a given application.

**Developing skill in expressing oneself orally or in writing**

- Students will be able record experimental data and results in a manner in which it can be read and understood by anyone having a basic knowledge of analytical chemistry and that will permit the experiment to be duplicated.
- Students will be able to summarize experimental results in concise reports that meet requirements of the task at hand and the intended recipient of the report.

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1 Analysis skills are required by chemists, biochemists, biologists, geologists, environmental scientists, medical researchers and technicians, food scientists, engineers, etc.
Student Responsibilities

Attendance
Class attendance is expected. The student is responsible for making-up any work missed due to absence from class. The student will only be permitted to make up missed work under the following conditions:

- Unavoidable emergency absences (illness, death in the immediate family, etc.): you must contact me during or before the class immediately following the absence with a valid, verifiable excuse.
- Planned absences (trips out of town, etc.): you must get my approval make arrangements to make up missed work prior to the absence. The reason for the absence should warrant missing class to get my approval: participation in University sponsored events would fall in this category.

Class Preparation
Read each reading assignment before it is covered in class; come to class prepared to discuss the material. Contribute to in-class discussions of the material.

Homework, Assignments and Quizzes
Do all assigned homework problems rather they are graded or not. Assignments from the text will typically not be graded as the answers are in the back of the text and the solutions manual is available to you in the bookstore. During the class that immediately follows the giving of an assignment, you may take the opportunity to ask questions of that assignment at the start of the class. After they are graded or discussed in class, make sure you understand them. Quizzes are worth 20 points and will cover material covered since the last quiz or exam. It is to your advantage to get an early start on each assignment and to review your class notes between lectures.

Exams
Demonstrate your knowledge and understanding of the material covered on exams. All exams are cumulative; however, regular exams will predominantly (usually > 90% of exam) cover material covered since the last exam. The Final Exam will be comprehensive.

Exams cannot be made-up, except in the following circumstances:
- Previous arrangements are made with me
- Serious illness (note from a physician, etc. required)
- Death in the immediate family

Laboratory
Laboratory work is an essential part of a science course. Students should make every effort to participate fully in the laboratory experience. Students should approach the lab as if it were a research project. Every effort should be made to obtain quality results, record the experimental work completely at the time the work is done, evaluate the quality of the work and take steps to improve results when needed and possible. Material from lab experiments may be covered on lecture exams. Both a formal report (produced using a word processor such as Word) and the lab notebook will be submitted and graded for each lab.

- **Major Lab Reports** (30 pts) for labs that require a more extensive discussion of data. These should have a format similar to a article in a scientific journal and will be 6-10 pages. Labs requiring a major lab report are indicated with an asterisk in the course schedule.
- **Minor Lab Reports** (15 pts) for all other labs will follow the format of an inter-lab report and will typically be 2-5 pages in length.
- **Lab Notebook spot checks.** Each Student will have a personal Lab Notebook group in
Blackboard. Students are to upload a complete scan of the current experiment at the following times.

- Before start of lab – should include introduction and procedure sections.
- End of the lab period – will also include all records recorded that day.
- Mid-lab ONLY IF REQUESTED BY INSTRUCTOR
- Time of lab report submission

Certain of these uploads will be checked unannounced and points may be awarded based on whether the upload was done at the appropriate time and the quality of the lab record at that point.

- **Final Lab Notebook Check.** At the end of the semester, lab notebooks will be collected and graded.
- At the end of a semester you will be evaluated by the lab instructor in regard to lab technique and safety practices. This evaluation will be worth 25 points.

**General Work Practices**

- Always show all work when doing mathematical problems on homework, on tests and in the lab.
- All numerical answers must be written with the correct number of significant digits and appropriate units
General Policies Related to This Course
All students are required to follow the policies and procedures presented in these documents:
- Angelo State University Student Handbook
- Angelo State University Catalog

Academic Integrity
Students are expected to maintain complete honesty and integrity in all work. Any student found guilty of any form of dishonesty in academic work is subject of disciplinary action and possible expulsion from ASU.

The College of Science and Engineering adheres to the university's Statement of Academic Integrity.

Accommodations for Students with Disabilities
ASU is committed to the principle that no qualified individual with a disability shall, on the basis of disability, be excluded from participation in or be denied the benefits of the services, programs or activities of the university, or be subjected to discrimination by the university, as provided by the Americans with Disabilities Act of 1990 (ADA), the Americans with Disabilities Act Amendments of 2008 (ADAAA) and subsequent legislation.

Student Disability Services is located in the Office of Student Affairs, and is the designated campus department charged with the responsibility of reviewing and authorizing requests for reasonable accommodations based on a disability. It is the student's responsibility to initiate such a request by contacting an employee of the Office of Student Affairs, in the Houston Harte University Center, Room 112, or contacting the department via email at ADA@angelo.edu. For more information about the application process and requirements, visit the Student Disability Services website. The employee charged with the responsibility of reviewing and authorizing accommodation requests is:

Dallas Swafford
Director of Student Disability Services
Office of Student Affairs
325-942-2047
dallas.swafford@angelo.edu
Houston Harte University Center, Room 112

Incomplete Grade Policy
It is policy that incomplete grades be reserved for student illness or personal misfortune. Please contact faculty if you have serious illness or a personal misfortune that would keep you from completing course work. Documentation may be required. See ASU Operating Policy 10.11 Grading Procedures for more information.
Plagiarism
Plagiarism is a serious topic covered in ASU’s Academic Integrity policy in the Student Handbook. Plagiarism is the action or practice of taking someone else’s work, idea, etc., and passing it off as one’s own. Plagiarism is literary theft.

In your discussions and/or your papers, it is unacceptable to copy word-for-word without quotation marks and the source of the quotation. It is expected that you will summarize or paraphrase ideas giving appropriate credit to the source both in the body of your paper and the reference list.

Papers are subject to be evaluated for originality. Resources to help you understand this policy better are available at the ASU Writing Center.

Student Absence for Observance of Religious Holy Days
A student who intends to observe a religious holy day should make that intention known in writing to the instructor prior to the absence. See ASU Operating Policy 10.19 Student Absence for Observance of Religious Holy Day for more information.

Title IX at Angelo State University
The University prohibits discrimination based on sex, which includes pregnancy, sexual orientation, gender identity, and other types of Sexual Misconduct. Sexual Misconduct is a broad term encompassing all forms of gender-based harassment or discrimination including: sexual assault, sex-based discrimination, sexual exploitation, sexual harassment, public indecency, interpersonal violence (domestic violence and/or dating violence), and stalking. As a faculty member, I am a Responsible Employee meaning that I am obligated by law and ASU policy to report any allegations I am notified of to the Office of Title IX Compliance.

Students are encouraged to report any incidents of sexual misconduct directly to ASU’s Office of Title IX Compliance and the Director of Title IX Compliance/Title IX Coordinator at:

Michelle Boone, J.D.
Director of Title IX Compliance/Title IX Coordinator
Mayer Administration Building, Room 210
325-942-2022
michelle.boone@angelo.edu

You may also file a report online 24/7 at www.angelo.edu/incident-form.

If you are wishing to speak to someone about an incident in confidence you may contact the University Health Clinic and Counseling Center at 325-942-2173 or the ASU Crisis Helpline at 325-486-6345.

For more information about Title IX in general you may visit www.angelo.edu/title-ix.
## Proposed Course Schedule

**CHEM 4421 — Instrumental Analysis — Spring 2019**

**Exam 1:** Thursday, Feb 20  
**Exam 2:** Tuesday, April 14  
**Final Exam:** Tues, May 5 8:00-10:00 AM

The last day to drop the course with a grade of "W" is Thurs, Mar. 26, 2019.

**Lab Due Dates:** unless indicated otherwise, lab reports are due 5:00 PM the Monday following the lab.

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<tr>
<th>Wk #</th>
<th>Lecture Topics</th>
<th>Lab</th>
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| 1    | Tue 1/14: Electrochemistry Review, Chap. 14  
Electrodes and Potentiometry, Chap. 15  
Thur 1/16: Electrolysis, Electrogravimetry, Coulometry  
Sec's 17.1-17.3 | Lab Thurs 1/16  
No Lab This Week  
Calibration Curves, Sec. 4-8, 4-9  
Required University Lab Safety Course due Monday 1/20 |
| 2    | Tues 1/21: Quiz. Figures of Merit, Sec. 5-1, 5-2  
Thur 1/23: Advanced Calibration Methods/Catch Up  
Homework: Advance Calibration Methods - Tutorial Projects  
• Standard Addition, Sec. 5-3;  
• Internal Standards, Sec. 5-4 | Lab Thurs 1/23  
Experiment 1: Calibration of micropipets and dilutions  
Introduction to Flame Atomic Absorption Spectrophotometry |
| 3    | Tues 1/28: Quiz Amperometry, Voltammetry, Stripping Analysis Sec's 17-4, 17-5  
Thur 1/30: Microelectrodes, Sec. 17-5;  
Electroanalytical Applications | Lab Thurs 1/30 – Experiment 2/3 Rotation  
Experiment 2: Figures of Merit for UV-Vis Analysis of Iron: Effect of derivatization reagents. (Submit lab notebook report ONLY)  
Experiment 3: Figures of Merit Atomic Adsorption Analysis (Submit lab notebook report ONLY) |
| 4    | Tues 2/4: Quiz Review Spectroscopy Basics, Sec's 18-1 to 18-4  
Thur 2/6: Sec's 18-6, 18-7 Classify Spectroscopies by  
• Region of spectrum  
• How light interacts with sample (absorption, emission, fluorescence, scattering, etc.)  
• What species can be analyzed | Lab Tues Thurs 2/6  
• Experiment 2/3 Rotation (Cont.)  
• Experiment 2/3 Combined Major Report: Comparison of the analysis of iron using UV-Vis and AA methods*. Due 5:00 PM Friday, Feb. 15 |
<p>| 5    | Tues 2/11: Quiz UV-Vis | Lab Thurs 2/13 – Experiment 4/5 Rotation |</p>
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<tr>
<th>Day</th>
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<tbody>
<tr>
<td>6</td>
<td>Tues 2/18</td>
<td>Spectrometers (cont)</td>
<td>Lab Thurs 2/20 – Experiment 4/5 Rotation (cont.)</td>
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<tr>
<td></td>
<td>Thur 2/20</td>
<td>Exam 1 (Sec. 4-8, Chap's 5, 15, 17 and 18)</td>
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<td>7</td>
<td>Tues 2/25</td>
<td>Fluorescence, Phosphoresce, Sec. 18-7</td>
<td>Lab Thurs 2/27 – Experiment 6</td>
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<td>Thur 2/27</td>
<td>Chemiluminescence, Scattering Methods, Sec. 18-7</td>
<td>Simultaneous Analysis of Two Ions, Sec. 19-1</td>
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<td>8</td>
<td>Tues 3/3</td>
<td>Quiz, X-ray methods Sec. 21-8</td>
<td>Lab Thurs 3/5 – Experiment 7</td>
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<td>Thur 3/5</td>
<td>Introduction to Mass Spectrometry (MS)</td>
<td>Spectrophotometric Determination of pKa of an Indicator</td>
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<td>March 9-13</td>
<td>Spring Break</td>
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<td>9</td>
<td>Tues 3/17</td>
<td>Quiz. Atomic Spectroscopy Intro 21-1</td>
<td>Lab Thurs 3/19 – Experiment 8/9 Rotation</td>
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<td>Atomic Absorption (AA)/ Atomic Emission (AE or OES), Sec’s 21-2 to 21-4</td>
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<td>Thur 3/19</td>
<td>Spectrometer Design for Atomic Spectroscopy</td>
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<td>Background Correction &amp; Interference, Sec’s 21-5, 21-6</td>
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<td>Tues 3/24</td>
<td>Quiz. Interferometers &amp; FTIR, Sec. 20-5</td>
<td>Lab Thurs 3/26 – Extra Credit for attending Moon Lecture</td>
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<td>Wrap Up Optical Spectroscopy</td>
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<td>Thur 3/26</td>
<td>ICP-MS, Sec. 21-7</td>
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<td>*** Thursday, Mar. 26 – Last Day to Drop ***</td>
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<td>10</td>
<td>Tues 3/31</td>
<td>Quiz. EI MS, Sec. 22-1; Mass Analyzers, Sec. 22-3</td>
<td>Lab Thurs 3/28 – Experiment 8/9 Rotation (cont.)</td>
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<td>Thur 4/2</td>
<td>Mass Analyzers (cont.)</td>
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<td>11</td>
<td>Tues 4/7</td>
<td>Quiz. MS in chromatography, Sec. 22-4, 22-5</td>
<td>Lab Thurs 4/2 Experiment 10</td>
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<td>Other MS Methods, Sec’s 22-6 &amp; 22-7</td>
<td>Mass Spectral Interpretation*, Sec. 22-2 (Due 5:00 PM April 10)</td>
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<td>Thur 4/9</td>
<td>Introduction to Separations, Sec’s 23-1, 23-2</td>
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<td>Lab Thurs 4/9: Experiment 11/12 Rotation</td>
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- **What species absorb (mechanisms)**
- **Advanced Applications**
- **Experiment 4**: Fluoride Ion Selective Electrodes - Manual Calibration
- **Experiment 5**: Cyclic voltammetry* (Due 5:00 PM 8 days after lab)
- **Experiment 6**: Spectrometer Design for Atomic Spectroscopy
- **Experiment 7**: Spectrophotometric Determination of pKa of an Indicator
- **Experiment 8**: Noise Reduction Methods (Lecture Project), Sec. 20-6
- **Experiment 9**: Introduction to Analytical Fluorescence* (Due 5:00 PM 8 days after lab)
- **Experiment 10**: Mass Spectral Interpretation*, Sec. 22-2 (Due 5:00 PM April 10)
- **Experiment 11**: Spectrophotometric Titration of Colored Metal Complex, Sec’s 18-5, 19-3
- **Experiment 12**: Analyzing GC-MS Data
<table>
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<th>Week</th>
<th>Tuesday</th>
<th>Thursday</th>
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| 13   | 4/14: Exam 2 (Chapters 18-22)  
**Thur 4/16:** Chromatography constraints, Sec's 23-3 to 23-5  
Gas chromatography (GC): columns and sample introduction, Sec's 24-1, 24-2 | Lab Thurs 4/16 Experiment 11/12 Rotation (coot.) |
| 14   | 4/21: GC Detectors, Sec. 24-3  
**GC Sample Prep and Applications, Sec. 24-4, 24-5** | Lab Thurs 4/23 Experiment 13  
Optimizing GC Conditions* (Due 5:00 PM May 1) |
| 15   | 4/28: Quiz. HPLC columns and methods, Sec. 25-2  
**Thur 4/30:** HPLC detectors and gradients, Sec's 25-2 to 25-4 | Thurs 4/30 - Check Out (Lab Final - Qualitative Analysis of Pizza, etc.) |
| Final| Tuesday May 5 Final Exam  
8:00-10:00 AM in CAV 223 | 40% Chapters 23-25, 60% Comprehensive |

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2. [https://www.angelo.edu/student-handbook/](https://www.angelo.edu/student-handbook/)  
3. [https://www.angelo.edu/catalogs/](https://www.angelo.edu/catalogs/)  
5. [https://www.angelo.edu/services/disability-services/](https://www.angelo.edu/services/disability-services/)  
6. [https://www.angelo.edu/content/files/14197-op-1011-grading-procedures](https://www.angelo.edu/content/files/14197-op-1011-grading-procedures)  
8. [https://www.angelo.edu/dept/writing_center/academic_honesty.php](https://www.angelo.edu/dept/writing_center/academic_honesty.php)  
9. [https://www.angelo.edu/content/files/14206-op-1019-student-absence-for-observance-of](https://www.angelo.edu/content/files/14206-op-1019-student-absence-for-observance-of)