Syllabus CHEM 4421
Instrumental Analysis - Spring 2019

Instructor
Dr. David Carter
Office hours: MW 1:30-3:00 PM, Th 9:30-11:30 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 8:00-9:15A.M.  CAV 223
Lab: Th 2:00-4:50 P.M., CAV 206/203   (Prelab: CAV 215)

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 that is 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 . . . >970
Sapling Homework (165 pts possible)                          150 pts     B . . . 840-980
Exams                                                   200 pts     C . . . 720-840
Lab (325 pts possible)                                       300 pts     D . . . 600-720
Final Exam                                                200 pts     F . . . <720
Total                                      1100 pts
<table>
<thead>
<tr>
<th>Student Learning Outcomes</th>
<th>Dept. SLO</th>
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<tbody>
<tr>
<td><strong>Essential Learning Outcomes</strong>&lt;br&gt;Note: letter notations in parentheses after each objective refer to the Student Learning Objectives of the Department of Chemistry and Biochemistry. These are available in the Course Information section of Blackboard.</td>
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<tr>
<td><strong>Learning to apply course material</strong>&lt;br&gt;(to improve thinking, problem solving, and decisions)</td>
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<td>- 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.</td>
<td>3b</td>
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<td>- Students should be able to apply the general principles of instrumental methods to understand and evaluate unfamiliar or new instrumental techniques.</td>
<td>4a</td>
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<td><strong>Developing specific skills and competencies</strong>&lt;br&gt;(needed by professionals in the fields most closely related to this course)</td>
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<td>- 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</td>
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<td>- Optimize instrument performance</td>
<td>3b</td>
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<td>- Process and analyze data</td>
<td>2b</td>
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<td>- Interpret data in a meaningful way (requires an awareness of method limitations)</td>
<td>1a</td>
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<td>- Understand at a technical level how instruments work</td>
<td>3b</td>
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<td><strong>Important Goals</strong></td>
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<td><strong>Gaining factual knowledge (terminology, classifications, methods, trends)</strong></td>
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<td>- Students will be able to identify and name the major instrumental methods.</td>
<td>3b</td>
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<td>- Students will be able to classify those methods according to the following criteria:</td>
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<td>- Type of method (spectroscopic, chromatographic, electrochemical, thermal, etc.)</td>
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<td>- Type of analyte</td>
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<tr>
<td>- element, compound, pure or mixture</td>
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<td>- solid, liquid or gas</td>
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<td>- bulk or surface</td>
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<tr>
<td>- Information required</td>
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<tr>
<td>- qualitative vs. quantitative</td>
<td></td>
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<tr>
<td>- elemental vs. molecular</td>
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<td>- physical properties</td>
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<td>- concentration range (major component, minor component, trace, etc.)</td>
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<td><strong>Learning fundamental principles, generalizations, or theories</strong></td>
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<td>- 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.</td>
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<td>- Students will be able to used quantitative figures of merit to evaluate the suitability of a method for a given application.</td>
<td>1a</td>
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<td><strong>Developing skill in expressing oneself orally or in writing</strong></td>
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<td>- 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.</td>
<td>3b</td>
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<td>- Students will be able to summarize experimental results in concise reports that meet the requirements to the task at hand and the intended recipient of the report.</td>
<td>2a</td>
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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

1. 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:
   a. 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.
   b. 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.

2. 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.

3. 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.

4. 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:
   a. Previous arrangements are made with me
   b. Serious illness (note from a physician, etc. required)
   c. Death in the immediate family

5. 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.

   a. 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.
   b. 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.
   c. Lab Notebook spot checks. Students may be selected at random. At that time a digital scan may be made of their lab notebook pages associated with the lab in progress and evaluated for completeness. If the student’s lab notebook is not up to date when inspected, a penalty of 10 points up to the full value of the lab will be assessed. Students lab notebooks may be checked at the following times
       i. Start of lab to check that the prelab has been done.
       ii. Mid-lab period to insure that all students are recording data in real time.
       iii. As the student leaves the lab. All students need to check with instructor before leaving lab to see if their lab notebook needs to be checked.
   d. 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.

6. You must show all work when doing mathematical problems on the homework, on tests and in the lab.

7. All numerical answers must be written with the correct number of significant digits and appropriate units
Honor Code / Academic Dishonesty
Angelo State University expects its students to maintain complete honesty and integrity in their academic pursuits. Students are responsible for understanding the Academic Honor Code, which is to be found in the Student Handbook (www.angelo.edu/cstudent). The penalty for ANY sort of dishonesty, cheating or plagiarism can range from a grade of zero on the assignment to an F in the course and disciplinary action as warranted in accordance with university guidelines. Don’t even consider it.

Disabilities
Persons with disabilities which may warrant academic accommodations must contact the Student Life Office, Room 112 University Center, in order to request and to implement academic accommodations.

Title IX at Angelo State University:
Angelo State University is committed to providing and strengthening an educational, working, and living environment where students, faculty, staff, and visitors are free from sex discrimination of any kind. Sex discrimination, sexual misconduct, public indecency, interpersonal violence, sexual assault, sexual exploitation, sexual harassment, and stalking are not tolerated at ASU. As a faculty member, I am a Responsible Employee meaning that I will report any allegations I am notified of to the Office of Title IX Compliance in order to connect students with resources and options in addressing the allegations reported. You are encouraged to report any incidents to ASU’s Office of Title IX Compliance and the Director of Title IX Compliance/Title IX Coordinator. You may do so by contacting:

Michelle Boone, J.D.
Director of Title IX Compliance/Title IX Coordinator
Mayer Administration Building, Room 200
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.

The Office of Title IX Compliance also provides accommodations related to pregnancy (such as communicating with your professors regarding medically necessary absences, modifications required because of pregnancy, etc.). If you are pregnant and need assistance or accommodations, please contact the Office of Title IX Compliance utilizing the information above.

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 21  Exam 2: Tuesday, April 16  Final Exam: Tues, May 7, 8:00-10:00 AM

The last day to drop the course with a grade of “W” is Thurs, Mar. 28, 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 1/15:** Electrochemistry Review, Chap’s 14-16  
Thur 1/17: Electrolysis, Electrogravimetry, Coulometry  
Sec’s 17.1-17.3 | Lab Thurs 1/17  
Lab Notebooks & Pre-Lab  
Calibration Curves, Sec. 4-8, 4-9  
Required University Lab Safety Course due Monday 1/22 |
| 2    | **Tues 1/22:** Quiz. Figures of Merit, Sec. 5-1, 5-2  
Thur 1/24: Advanced Calibration Methods/Catch Up  
*Homework:* Advance Calibration Methods - Tutorial Projects  
Standard Addition, Sec. 5-3 and Internal Standards, Sec. 5-4 | Lab Thurs 1/24  
**Experiment 1:** Calibration of micropipets and dilutions  
Introduction to Flame Atomic Absorption Spectrophotometry |
| 3    | **Tues 1/29:** Quiz Amperometry, Voltammetry, Stripping Analysis  
Sec’s 17-4, 17-5  
Thur 1/31: Microelectrodes, Sec. 17-5; Electroanalytical Applications | Lab Thurs 1/31  
**Experiment 2A:** Figures of Merit for UV-Vis Analysis of Iron: Effect of derivatization reagents. (Submit lab notebook report ONLY) |
| 4    | **Tues 2/5:** Quiz Review Spectroscopy Basics, Sec’s 18-1 to 18-4  
Thur 2/7: Sec’s 18-6, 18-7 Classifying Spectroscopies by  
- Region of spectrum  
- How light interacts with sample (absorption, emission, fluorescence, scattering, etc.)  
- What species can be analyzed | Lab Tues Thurs 2/7  
**Experiment 2B: Figures of Merit** Atomic Adsorption Analysis (Submit lab notebook report ONLY) |
| 5    | **Tues 2/12:** Quiz UV-Vis  
- What species absorb (mechanisms)  
- Advanced Applications  
Thur 2/14: Spectrometers (UV-Vis focus) Sec’s 20-1 to 20-3 | Lab Thurs 2/14  
**Rotation 1:**  
- Exper. Rot-1A: Fluoride Ion Selective Electrodes - Manual Calibration  
- Exper. Rot-1B: Simultaneous Analysis of Two Ions, Sec. 19-1  
- Exper. Rot-1C: Spectrophotometric Determination of pKa of an Indicator  
- Experiment Rot-1D: Cyclic voltammetry* (Due 5:00 PM 8 days after lab) |
| 6    | **Tues 2/19:** Spectrometers (cont)  
Thur 2/21: Exam 1 (Sec. 4-8, Chap’s 5, 15, 17 and 18 | Lab Tues 2/20 & Thurs 2/22 - Rotation 1 (cont.) |
| 7    | **Tues 2/26:** Fluorescence, Phosphoresce, Sec. 18-7  
Thur 2/28: Chemiluminescence, Scattering Methods, Sec. 18-7 | Lab Thurs 3/2 - Rotation 1 (cont.) |
<table>
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<tr>
<th>Week</th>
<th>Dates</th>
<th>Monday</th>
<th>Tuesday</th>
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<th>Thursday</th>
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| 8    | 3/5-3/7     | Quiz. X-ray methods Sec. 21-8  
Thur 3/7: Introduction to Mass Spectrometry (MS) |               |               | Lab Thurs 3/7 - Rotation 1 (cont.) |
| 9    | 3/10-3/14   | Spring Break  
March 11-15: Spring Break |               |               | Lab Thurs 3/11: Rotation 2 (cont.)  
- Exper. Rot-2A: Noise Reduction Methods (Lecture Project), Sec. 20-6  
- Exper. Rot-2B: Spectrophotometric Titration of Colored Metal Complex, Sec’s 18-5, 19-3  
- Exper. Rot-2C: Analyzing GC-MS Data  
- Exper. Rot-2D: Experiment Rot-2A: Introduction to Analytical Fluorescence* (Due 5:00 PM 8 days after lab) |
Atomic Absorption (AA)/ Atomic Emission (AE or OES), Sec’s 21-2 to 21-4  
Thur 3/21: Spectrometer Design for Atomic Spectroscopy  
Background Correction & Interference, Sec’s 21-5, 21-6 |               |               | Tues 3/26 - Extra Credit for attending Moon Lecture  
Lab Thurs 3/26 Rotation 2 (cont.) |
| 11   | 3/24-3/28   | Quiz. Interferometers and FTIR, Sec. 20-5  
Wrap Up Optical Spectroscopy  
Thur 3/28: ICP-MS, Sec. 21-7  
*** Thursday, Mar. 28 - Last Day to Drop *** |               |               | Lab Thurs 4/4 Rotation 2 (cont.) |
| 12   | 3/31-4/4    | Quiz. El MS, Sec. 22-1; Mass Analyzers, Sec. 22-3  
Thur 4/4: Mass Analyzers (cont.) |               |               | Lab Thurs 4/11: Rotation 2 (cont.) |
| 13   | 4/5-4/9     | Quiz. MS in chromatography, Sec. 22-4, 22-5  
Thur 4/11: Other MS Methods, Sec’s 22-6 & 22-7  
Introduction to Separations, Sec’s 23-1, 23-2 |               |               | Lab Thurs 4/18  
Mass Spectral Interpretation*, Sec. 22-2 (Due 5:00 PM April 24) |
| 14   | 4/10-4/14   | Quiz. GC Detectors, Sec. 24-3  
GC Sample Prep and Applications, Sec. 24-4, 24-5  
Thur 4/18: Chromatography constraints, Sec’s 23-3 to 23-5  
Gas chromatography (GC): columns and sample introduction, Sec’s 24-1, 24-2 |               |               | Lab Thurs 4/25  
Optimizing GC Conditions* (Due 5:00 PM May 1) |
| 15   | 4/15-4/19   | Quiz. HPLC columns and methods, Sec. 25-2  
Thur 5/2: HPLC detectors and gradients, Sec’s 25-2 to 25-4 |               |               | Thurs 5/2 – Check Out (Lab Final – Qualitative Analysis of Pizza, etc.) |
| Final| 4/22-5/1    |               |               | Tuesday May 7 Final Exam  
8:00-10:00 AM in CAV 223 |               | 40% Chapters 23-25, 60% Comprehensive |

* Due to time constraints, please ensure all assignments are submitted before the due dates.