Instrumental Analysis - CHEM 3418
Department of Physical and Environmental Sciences
Spring 2020

A. COURSE INFORMATION

Course number/section: CHEM 3418.001 lecture; labs CHEM 3418.101, 102, and 103
Class meeting time: Lecture (TR 11:00-12:15) Labs (101 – T 8:00-10:50; 102 – T
12:30-3:20; 103 - R 8:00-10:50)
Class location: Lecture – CS 101, Labs CS 221
Course Website: https://bb9.tamucc.edu/

B. INSTRUCTOR INFORMATION

Instructor: Dr. Eugene Billiot
Office location: CS 208
Office hours: Tuesday and Thursday 3:30 – 5:00 and Monday 10:00-12:00
Telephone: (361) 825-2680
e-mail: Eugene.Billiot@tamucc.edu
Appointments: email me to schedule appointments outside of office hours

C. COURSE DESCRIPTION

Catalog Course Description
An introduction to instrumental methods of analysis: spectroscopy, chromatography, and
electrochemical methods. Laboratory involves use of instrumentation in chemical analysis.
Prerequisite: CHEM 1412. Safety training given during a laboratory meeting early in the
semester is required for continued participation in this course.

Extended Course Description
In this course, students will learn basic molecular and atomic spectroscopy and
chromatography instrumentation.

D. PREREQUISITES AND COREQUISITES

Prerequisites
CHEM 1412, and CHEM 3412
Corequisites
SMTE0093

E. REQUIRED TEXTBOOK(S), READINGS AND SUPPLIES

Required Textbook(s)
Principles of Instrumental Analysis, 6th edition or later edition. Skoog, Holler, and Crouch,

Optional Textbook(s) or Other References
Supplies

Proper lab attire should be worn in lab.

F. STUDENT LEARNING OUTCOMES AND ASSESSMENT

Assessment is a process used by instructors to help improve learning. Assessment is essential for effective learning because it provides feedback to both students and instructors. A critical step in this process is making clear the course’s student learning outcomes that describe what students are expected to learn to be successful in the course. The student learning outcomes for this course are listed below. By collecting data and sharing it with students on how well they are accomplishing these learning outcomes students can more efficiently and effectively focus their learning efforts. This information can also help instructors identify challenging areas for students and adjust their teaching approach to facilitate learning.

After completion of this course, the student will have an increased understanding of various analytical tools (instruments) utilized by scientists to obtain qualitative and quantitative information about the composition and structure of matter. The laboratory portion of this course is designed to reinforce the concepts being taught in the lecture portion. This will be achieved through practical hands-on experience with the techniques and instruments used in modern analytical laboratories, as well as peer-mentored learning. By the end of this course, students should be able to:

1. The student will gain an understanding of the advantages and limitations of various analytical tools so that they can choose the most appropriate instrumental method and be attuned to its limitations in terms of sensitivity, precision and accuracy.
2. The student will also gain knowledge of measurement principles in order to be able to make informed decisions about calibration, standardization and validation of instrumental methods.

G. INSTRUCTIONAL METHODS AND ACTIVITIES

<table>
<thead>
<tr>
<th>TESTS: 3 @ 100 pts each</th>
<th>300 pts</th>
</tr>
</thead>
<tbody>
<tr>
<td>These tests are cumulative with emphasis on material covered since the last test. The format of the tests are to be determined. The test may consist of problems which must be set up and solved, multiple choice, fill in the blank, matching, and/or discussion questions, and terms to be defined.</td>
<td>75% of your total grade</td>
</tr>
<tr>
<td><strong>Make up policy:</strong> No make-up tests will be given. If you have an excused absence for one of the tests AND with approval of the instructor, your final exam grade will be substituted for the missed test. If you do not miss any tests, your lowest test score will be replaced with your final exam grade, provided that your final exam score is higher.</td>
<td></td>
</tr>
<tr>
<td><strong>LAB GRADE:</strong> see lab syllabus</td>
<td>100 pts</td>
</tr>
</tbody>
</table>
**FINAL EXAM:** The final exam is comprehensive. It will replace your lowest test score (if it is higher than the lowest test score) or serve as a makeup exam if you missed a test.

**TOTAL POINTS**

<table>
<thead>
<tr>
<th>ACTIVITY</th>
<th>% of FINAL GRADE</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lecture - exams</td>
<td>75%</td>
</tr>
<tr>
<td>Lab</td>
<td>25%</td>
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</tbody>
</table>

**H. MAJOR COURSE REQUIREMENTS AND GRADING**

As discussed previously, three regular semester exams and a comprehensive final exam will be given in the lecture. The lowest of those four grades will be dropped and the remaining three grades will be averaged to calculate your grade in the lecture portion of the course. The lab grade which will be 25% of your overall course grade will be determined based on lab reports and/or quizzes and possibly a lab final exam.

**TOPICS**

<table>
<thead>
<tr>
<th>Week of</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chapter 1, Appendix 1, &amp; Chapter 6</td>
</tr>
<tr>
<td>Chapters 6 &amp; 7</td>
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<tr>
<td>Chapters 7 &amp; 8</td>
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<tr>
<td>Chapters 9 &amp; 10 (maybe some of Chapters 11 &amp; 12)</td>
</tr>
<tr>
<td>Review and Exam 1</td>
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<tr>
<td>Chapter 13</td>
</tr>
<tr>
<td>Chapter 14</td>
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<tr>
<td>Chapters 15 and 16</td>
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<tr>
<td>Chapter 20</td>
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<tr>
<td>Spring Break</td>
</tr>
<tr>
<td>Review and Exam 2</td>
</tr>
<tr>
<td>Chapter 26</td>
</tr>
<tr>
<td>Chapter 26 and 27</td>
</tr>
<tr>
<td>Chapter 27 and 28</td>
</tr>
<tr>
<td>Chapter 28 and 30</td>
</tr>
<tr>
<td>Chapter 30 and Review for Exam 3</td>
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<tr>
<td>Exam 3</td>
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</tbody>
</table>
Note: Changes in this course schedule may be necessary and will be announced to the class by the Instructor. The assignments and exams shown are directly related to the Student Learning Outcomes described in Section F.

J. COURSE POLICIES

Attendance/Tardiness
Absences and tardiness will not be tolerated. It is disrespectful to the instructor and to the students when you show up late. Five points will be deducted from your grade for any unexcused absences or tardiness. If you must be absent or late please let the instructor know ahead of time. (NOTE: You have my email address and my cell phone number [See Blackboard site for this course]. There should be no excuse for not informing me if you have to be late or absent.)

Late Work and Make-up Exams
No make-up tests will be given. If you have an excused absence for one of the tests AND with approval of the instructor, your final exam grade will be substituted for the missed test. If you do not miss any tests, your lowest test score will be replaced with your final exam grade, provided that your final exam score is higher.

Extra Credit
none

Cell Phone Use
Please turn off cell phones and do not use during lecture or lab.

Laptop Use
Allowed for note taking but not during an exam.

Food in Class
Allowed but please be respectful of others.

Missed Exam
As mentioned above, no makeup exams will be given. If you know ahead of time that you will not be present during the regularly scheduled exam time please arrange a time to take the test ahead of time.

Participation
All students are expected to treat the other students and the instructor with respect. If a student's action or behavior is deemed disruptive by the instructor, the student will be asked to leave for that day. If the behavior persists, further disciplinary action may be taken, such as expulsion from the class and failure of the course.

Others
Academic Integrity/Plagiarism*

University students are expected to conduct themselves in accordance with the highest standards of academic honesty. Academic misconduct for which a student is subject to penalty includes all forms of cheating, such as illicit possession of examinations or examination materials, falsification, forgery, complicity or plagiarism. (Plagiarism is the presentation of the work of another as one’s own work.) In this class, academic misconduct or complicity in an act of academic misconduct on an assignment or test will result in a minimum of a F on the assignment but depending on the situation it may result in a failing grade (F) in this class, not simply the assignment.

K. COLLEGE AND UNIVERSITY POLICIES

- Academic Integrity (University)
  University students are expected to conduct themselves in accordance with the highest standards of academic honesty. Academic misconduct for which a student is subject to penalty includes all forms of cheating, such as illicit possession of examinations or examination materials, falsification, forgery, complicity or plagiarism. (Plagiarism is the presentation of the work of another as one’s own work.) In this class, academic misconduct or complicity in an act of academic misconduct on an assignment or test will result in a failing grade.

- Classroom/Professional Behavior
  Texas A&M University-Corpus Christi, as an academic community, requires that each individual respect the needs of others to study and learn in a peaceful atmosphere. Under Article III of the Student Code of Conduct, classroom behavior that interferes with either (a) the instructor’s ability to conduct the class or (b) the ability of other students to profit from the instructional program may be considered a breach of the peace and is subject to disciplinary sanction outlined in article VII of the Student Code of Conduct. Students engaging in unacceptable behavior may be instructed to leave the classroom. This prohibition applies to all instructional forums, including classrooms, electronic classrooms, labs, discussion groups, field trips, etc.

- Statement of Civility
  Texas A&M University-Corpus Christi has a diverse student population that represents the population of the state. Our goal is to provide you with a high quality educational experience that is free from repression. You are responsible for following the rules of the University, city, state and federal government. We expect that you will behave in a manner that is dignified, respectful and courteous to all people, regardless of sex, ethnic/racial origin, religious background, sexual orientation or disability. Behaviors that infringe on the rights of another individual will not be tolerated.

- Deadline for Dropping a Course with a Grade of W (University)
  I hope that you never find it necessary to drop this or any other class. However, events
can sometimes occur that make dropping a course necessary or wise. Please consult with your academic advisor, the Financial Aid Office, and me, before you decide to drop this course. Should dropping the course be the best course of action, you must initiate the process to drop the course by going to the Student Services Center and filling out a course drop form. Just stopping attendance and participation WILL NOT automatically result in your being dropped from the class. Please consult the Academic Calendar (http://www.tamucc.edu/academics/calendar/) for the last day to drop a course.

- Grade Appeals (College of Science and Engineering)
  As stated in University Procedure 13.02.99.C2.01, Student Grade Appeal Procedures, a student who believes that he or she has not been held to appropriate academic standards as outlined in the class syllabus, equitable evaluation procedures, or appropriate grading, may appeal the final grade given in the course. The burden of proof is upon the student to demonstrate the appropriateness of the appeal. A student with a complaint about a grade is encouraged to first discuss the matter with the instructor. For complete details, including the responsibilities of the parties involved in the process and the number of days allowed for completing the steps in the process, see University Procedure 13.02.99.C2.01, Student Grade Appeal Procedures. These documents are accessible through the University Rules website at http://www.tamucc.edu/provost/university_rules/index.html, and the College of Science and Engineering Grade Appeals webpage at http://sci.tamucc.edu/students/GradeAppeal.html. For assistance and/or guidance in the grade appeal process, students may contact the chair or director of the appropriate department or school, the Office of the College of Science and Engineering Dean, or the Office of the Provost.

- Disability Services
  The Americans with Disabilities Act (ADA) is a federal anti-discrimination statute that provides comprehensive civil rights protection for persons with disabilities. Among other things, this legislation requires that all students with disabilities be guaranteed a learning environment that provides for reasonable accommodation of their disabilities. If you believe you have a disability requiring an accommodation, please call (361) 825-5816 or visit Disability Services in Corpus Christi Hall 116.

  If you are a returning veteran and are experiencing cognitive and/or physical access issues in the classroom or on campus, please contact the Disability Services office for assistance at (361) 825-5816.

  http://disabilityservices.tamucc.edu/

- Statement of Academic Continuity*
  In the event of an unforeseen adverse event, such as a major hurricane and classes could not be held on the campus of Texas A&M University–Corpus Christi; this course would continue through the use of Blackboard and/or email. In addition,
the syllabus and class activities may be modified to allow continuation of the course. Ideally, University facilities (i.e., emails, web sites, and Blackboard) will be operational within two days of the closing of the physical campus. However, students need to make certain that the course instructor has a primary and a secondary means of contacting each student.

L. OTHER INFORMATION

• Academic Advising
The College of Science & Engineering requires that students meet with an Academic Advisor as soon as they are ready to declare a major. The Academic Advisor will set up a degree plan, which must be signed by the student, a faculty mentor, and the department chair. Meetings are by appointment only; advisors do not take walk-ins. Please call or stop by the Advising Center to check availability and schedule an appointment. The College’s Academic Advising Center is located in Center for Instruction 350 or can be reached at (361) 825-3928.

• How Course Information will be Relayed to Students:
Note that Blackboard will be used to relay all pertinent course information to the students. Therefore it is imperative that you (the student) check Blackboard on a regular basis to access information such as Study Guides, Power Point slides, etc. In addition it is important to note that I will be using the Blackboard email system to communicate with you about last minute assignment changes, etc so it is strongly advisable that you update your Blackboard account to include forwarding of mail messages to your preferred email account so that you can receive messages without having to log into blackboard.

GENERAL DISCLAIMER
I reserve the right to modify the information, schedule, assignments, deadlines, and course policies in this syllabus if and when necessary. I will announce such changes in a timely manner during regularly scheduled lecture periods.
Syllabus Information for Lab

TEXTBOOK AND OTHER REQUIRED MATERIALS:
Text: none
Lab: A lab coat and impact-resistant, splash-resistant goggles are required.

SEMESTER LABORATORY SCHEDULE (Tentative)

<table>
<thead>
<tr>
<th>Laboratory Experiments</th>
<th>Week of</th>
</tr>
</thead>
<tbody>
<tr>
<td>Introduction to Instrumentation</td>
<td>1-7</td>
</tr>
<tr>
<td><strong>Elemental Analysis experiments begin</strong></td>
<td>1-14</td>
</tr>
<tr>
<td>Elemental Analysis (continues)</td>
<td>1-21</td>
</tr>
<tr>
<td>Elemental Analysis (continues)</td>
<td>1-28</td>
</tr>
<tr>
<td>Elemental Analysis (continues)</td>
<td>2-4</td>
</tr>
<tr>
<td><strong>Molecular Spectroscopy experiments begin</strong></td>
<td>2-11</td>
</tr>
<tr>
<td>Molecular Spectroscopy (continues)</td>
<td>2-18</td>
</tr>
<tr>
<td>Molecular Spectroscopy (continues)</td>
<td>2-25</td>
</tr>
<tr>
<td><strong>Spring Break</strong></td>
<td>3-4</td>
</tr>
<tr>
<td>Molecular Spectroscopy (continues)</td>
<td>3-11</td>
</tr>
<tr>
<td><strong>Chromatography experiments begin</strong></td>
<td>3-18</td>
</tr>
<tr>
<td>Chromatography (continues)</td>
<td>3-25</td>
</tr>
<tr>
<td>Chromatography (continues)</td>
<td>4-1</td>
</tr>
<tr>
<td>Chromatography (continues)</td>
<td>4-8</td>
</tr>
<tr>
<td>Chromatography (continues)</td>
<td>4-15</td>
</tr>
<tr>
<td>Chromatography (continues)</td>
<td>4-22</td>
</tr>
<tr>
<td>Last week, no lab</td>
<td>4-29</td>
</tr>
</tbody>
</table>

ABSENCES and TARDINESS: As with lecture, absences and tardiness will not be tolerated. It is disrespectful to the instructor and to the students when you show up late. If you are late 5 points will be deducted from your grade. If you must be absent please let the instructor know ahead of time otherwise, as with tardiness, 5 points will be deducted from your grade. (NOTE: You have my email address and my cell phone number. There should be no excuse for not informing me if you have to be late or absent.)

GRADE ASSIGNEMENT: The grade for the laboratory portion of this course will be evenly distributed between three quizzes and three lab reports. **The lab reports are due at the beginning of the lab period in which they are due (see schedule). 25% will be deducted each day they are late. Note that if it is not given at the beginning of the lab period in which it is due that is counted as being a day late and thus 25% will be deducted from the grade.**

GRADING SCALE: The grades will be determined using the normal 10 point scale, i.e. 100-90 % A, 89-80 % B, 79-70 % C, 69-60 % D, and below 60 % F.

Remember that safety and good housekeeping / aka lab cleanup is very important. Unsafe behavior or poor housekeeping WILL negatively affect your grade. If proper lab safety and
procedures are not followed, points will be deducted from your grade. The amount of points deducted will be as follows:
  o first offense - there will be a warning
  o second offense – 10 points will be deducted from your grade
  o third offense – 50 points will be deducted from your lab grade
  o fourth offense – 100 points will be deducted from your grade and probable dismissal from the course

Some examples of safety violations and poor laboratory procedures include but are not limited to:
  • Not wearing proper safety equipment (gloves, safety glasses, lab coat and proper lab attire)
  • Eating or drinking in lab
  • Horseplay in lab
  • Pipetting or removing samples directly from stock bottles
  • Failure to clean and pick up all glassware and supplies utilized for that days experiment
  • Failure to clean balance after each use

CHEATING POLICY: Cheating in any form will not be tolerated. The University’s cheating and plagiarism policy as stated in the General Academic Policies and Regulations: Academic Honesty will be followed. Although group interactions, investigations and studying are encouraged, duplicative work will be treated as cheating and will receive a grade of zero. Even though you may be asked to work in pairs in the lab, each person is responsible for turning in a separate and unique lab report. Anything that is viewed as cheating on an exam will be given the most severe penalty possible. This will most likely include receiving an F in the course, and possibly other more severe punishments.

LAB CONDUCT: All students are expected to follow proper classroom conduct and behavior and treat the other students and the instructor with respect. If a student’s actions or behavior is deemed disruptive to the class by the instructor, the student will be asked to leave the class for that day. If the behavior persists, further disciplinary action may be taken, such as expulsion from the class. ABSOLUTELY no food or drink is allowed in the laboratory. Students that find it necessary to eat or drink in the lab or behave in some fashion that is deemed by the instructor to be unsafe to themselves or others will be asked to leave lab for that day and will be given a grade of zero for that assignment.

The "LAB REPORT"

Each lab must be typed or computer generated.
Hand written reports will not be accepted!!!!!!!!!!

Any copying or plagiarism from a text or a person will result in a Zero for that lab report. Text must be double spaced!

At the top any report should be the date, section number and your name. Your lab partner and
the unknown number (if there is one). The experiment name and number; i.e.

**Student:** Joe Bob Whissletip  
**Date:** April 15, 1998

**Partner:** Monica Clinton  
**Section:** 1

**Unknown number:** X-69  
**Group #:** 6

Experiment 22  
Determination of Biomolecule Unknown

These formal reports should contain the following information.

**Purpose**- composed of two or three sentences summarizing the experiment to be performed.

Example:
The purpose of this experiment is to determine the concentration of the protein using a standard curve derived from the UV/VIS spectrophotometer. Its molecular weight is determined using size exclusion chromatography.

**Abstract:** A brief summary (1-3 paragraphs describing what was done and why)

**Theory**- the length is not the most important consideration, but must contain:

1) A description of the theory which is being used in the experiment  
2) Application of the theory to your system  
3) Include all chemical equations  
4) Description of the instrument if one is used  
5) Diagram of instrument (boxes are OK just label the parts)  
6) Type of information obtained from instrument  
7) Brief description of the data analysis or processing of the data  
8) Expected results

**Procedure and observations:**

Tell how and what steps you followed as if you were the first one to ever do them. This can be as a narrative or bulleted/numbered format.

**Results and Discussion:** composed of:

1) Data in tables and instrumental data output should be attached permanently to a sheet of paper.

2) Graphs, properly labeled, are to be **computer generated.** There will be no hand drawn graphs. They are not to be photocopied from your partner.
3) Calculations: **GIVE AT LEAST ONE EXAMPLE!!!!!.** This should be well documented with a description of how you did these at each step and why you did what you did.

4) Since **all experiments are done in at least triplicate** there should always be a **mean**, **average** and **standard deviation**.

5) Final answers should be well labeled with the appropriate units and with correct **significant figures**. (Yes these count here in the lab).

6) Intelligibly communicate a subjective evaluation of:

- Your own execution of the experiment,
- The performance of the instruments and materials used
- The validity of the data collected and the phenomena observed; This means compare it to the literature values.
- Clearly state your results,
  i.e. The unknown copper solution, sample 5A, was found to be 5 ppm copper.

4. On the procedure you have either made a measurement or manipulated the data mathematically or observed a physical property or a combination of the above, **therefore ---- Ask yourself ---- ?**

- Is this reasonable?
- Does it Coincide with theory?
- Can I justify it?
- For unreasonable data ask **WHY**?
  Did the apparatus \ instrument fail?,
  Did I ......
  • make a mistake?,
  • misread the procedure?,
  • perform the experiment improperly?

Were chemicals contaminated?,
Are the instructions incomplete?

5. Suggest improvements that could address the short comings ascertained from your evaluations, in (4).

6. Be sure to write in complete sentences and in scientific language, be concise.

*7. If still unsure about how to write the report, then read any scientific research journal. ~ *Analytical Chemistry*, **Use it as a guide.**
Conclusions:
Briefly summarize lessons learned, as well as results achieved. Note that often when someone is reading scientific papers the two most important sections are the abstract and the conclusions sections. These two sections should have all of the info necessary to understand what was done and why and what were the results of the experiment as well as a summary of “lessons learned”.

References:
Give and note in your report's text!

Questions:
Answer them if they are given in the lab handouts.

THE LABORATORY NOTEBOOK:

1. ALL DATA IS TO BE RECORDED IN BLACK INK DIRECTLY IN THE NOTEBOOK!!!!
2. Notebooks should be bound and numbered.
3. Label and date entries.
4. An error should be lined through with a single horizontal line, initialed and dated.

Note: Begin each experiment on a new page.

Notebook Format

Title
Give the title of the experiment and the date at the top of the page for a new experiment.

Abstract
Give a brief description (a few sentences) of the experiment.

Procedure
Write a step by step description of what you do in the lab. Important chemical reactions should be included. Number your steps.

Data
Enter and tabulate data as it is collected. Record any observations or problems.