Instrumental Analysis - CHEM 3418
Department of Physical and Environmental Sciences
Spring 2015

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 – TBA; 103 – R 8:00-10:50)
Class location: Lecture – EN 107, Labs CS 221
Course Website: https://bb9.tamucc.edu/

B. INSTRUCTOR INFORMATION

Instructor: Dr. Eugene Billiot
Office location: CS 130
Office hours: TBD
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)

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

| TESTS: | 3 @ 100 pts each | 300 pts |
|        | 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. | 75 % of your total grade |
| Make up policy: 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 score, provided that your final exam score is higher. |
| LAB GRADE: see lab syllabus | 100 pts |
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>
</tr>
</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.

I. COURSE CONTENT/SCHEDULE

<table>
<thead>
<tr>
<th>TOPICS</th>
<th>Week of</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chapter 1, Appendix 1, &amp; Chapter 6</td>
<td>1-19</td>
</tr>
<tr>
<td>Chapters 6 &amp; 7</td>
<td>1-26</td>
</tr>
<tr>
<td>Chapters 7 &amp; 8</td>
<td>2-2</td>
</tr>
<tr>
<td>Chapters 9 &amp; 10 (maybe some of Chapters 11 &amp; 12)</td>
<td>2-9</td>
</tr>
<tr>
<td>Review and Exam 1</td>
<td>2-16</td>
</tr>
<tr>
<td>Chapters 13 &amp; 14</td>
<td>2-23</td>
</tr>
<tr>
<td>Chapters 15 &amp; 16</td>
<td>3-2</td>
</tr>
<tr>
<td>SPRING BREAK</td>
<td>3-9</td>
</tr>
<tr>
<td>Chapters 17 &amp; 18</td>
<td>3-16</td>
</tr>
<tr>
<td>Chapters 19 &amp; 20</td>
<td>3-23</td>
</tr>
<tr>
<td>Review and Exam 2</td>
<td>3-30</td>
</tr>
<tr>
<td>Chapter 26</td>
<td>4-6</td>
</tr>
<tr>
<td>Chapter 27</td>
<td>4-13</td>
</tr>
<tr>
<td>Chapter 28</td>
<td>4-20</td>
</tr>
<tr>
<td>Chapter 29 and Review for Exam 3</td>
<td>4-27</td>
</tr>
<tr>
<td>Exam 3</td>
<td>5-5</td>
</tr>
</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)
  It is expected that university students will demonstrate a high level of maturity, self-direction, and ability to manage their own affairs. Students are viewed as individuals who possess the qualities of worth, dignity, and the capacity for self-direction in personal behavior. See Full University Policy at [http://catalog.tamucc.edu/content.php?catoid=10&navoid=313#Academic_Integrity](http://catalog.tamucc.edu/content.php?catoid=10&navoid=313#Academic_Integrity)

- 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)
  The grade of W will be assigned to any student officially dropping a course by Friday, April 10, 2015. No student is eligible to receive a W without completing the official drop process by this deadline. Visit the Office of the University Registrar
for the Course Drop Form that must submitted. After April 10, 2015 a student will not be allowed 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](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](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**
  Disability Services (DS) is the hub for coordinating services and accommodations to ensure accessibility and utilization of all programs for all Texas A&M University-Corpus Christi students with disabilities. Our services are designed to meet the unique educational needs of enrolled students with documented permanent or temporary disabilities. DS provides intake and consultation services to students seeking to register with our office. DS reviews an individual’s documentation of disability and assesses eligibility for services and the determination of reasonable accommodations. For more information visit the Disability Services Office at 116 Corpus Christi Hall or go to [http://disabilityservices.tamucc.edu/](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

**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.
**Textbook and Other Required Materials:**

Text: *none*

Lab: A lab coat and impact-resistant, splash-resistant goggles are *required*.

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**Semester Laboratory Schedule (Tentative)**

**Laboratory Experiments**

<table>
<thead>
<tr>
<th>Week of</th>
<th>Laboratory Experiments</th>
</tr>
</thead>
<tbody>
<tr>
<td>1-19</td>
<td>None – short week</td>
</tr>
<tr>
<td>1-26</td>
<td>Introduction to Instrumentation</td>
</tr>
<tr>
<td>2-2</td>
<td><strong>Elemental Analysis experiments begin</strong></td>
</tr>
<tr>
<td>2-9</td>
<td>Elemental Analysis (continues)</td>
</tr>
<tr>
<td>2-16</td>
<td>Elemental Analysis (continues)</td>
</tr>
<tr>
<td>2-23</td>
<td>Elemental Analysis (continues)</td>
</tr>
<tr>
<td>3-2</td>
<td><strong>Molecular Spectroscopy experiments begin</strong></td>
</tr>
<tr>
<td>3-9</td>
<td>Spring Break</td>
</tr>
<tr>
<td>3-16</td>
<td>Molecular Spectroscopy (continues)</td>
</tr>
<tr>
<td>3-23</td>
<td>Molecular Spectroscopy (continues)</td>
</tr>
<tr>
<td>3-30</td>
<td><strong>Chromatography experiments begin</strong></td>
</tr>
<tr>
<td>4-6</td>
<td>Chromatography (continues)</td>
</tr>
<tr>
<td>4-13</td>
<td>Chromatography (continues)</td>
</tr>
<tr>
<td>4-20</td>
<td>Chromatography (continues)</td>
</tr>
<tr>
<td>4-27</td>
<td>Chromatography (continues)</td>
</tr>
</tbody>
</table>

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**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 Assignment:** 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:

- first offense - there will be a warning
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 day’s 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.
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.

**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. **NO cookbook** sentences in the typed report. Use both the passive and active voice, and you can use first person.

i.e. -----We stirred the solution for 10 min and decanted the liquid through a Whatman # 23 filter. The solution was then boiled for an additional 5 sec. After the boiling period, etc. ------

And NOT!! --- 1. Stir solution for 10 min.
2. Filter the solution.

**Results** - 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. **Do NOT forget to propagate your error or uncertainty. If you have not had Quantitative Analysis see me for information about propagation of error calculations.**

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).

**Discussion and Conclusion:**

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.**

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

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

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**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.
Lab Report Grading Guidelines  
(General Format to be used unless stated otherwise)

Presentation: Is the lab report neat in appearance, or is it just "thrown together?" The lab report must be stapled! (10 pts)

Format: Typed or generated by word processor: (30 pts)
On the whole, the report should contain:
- Correct heading as described in the examples
- Title
- Content
  Is the report well organized < ---- vs ----> poorly organized?
  Is it readable? Well-written?
  Are the objectives clearly stated? Does the discussion demonstrate what was done and give an indication about the results obtained.
  Reports should be written in complete sentence in your own words.
  Reports should be concise with correct spelling and grammatical usage.
- Discussion: Discuss your data, graphs, references, figures, error sources, etc.
- Summary
- Computer-generated graphs that are clearly labeled on both axes (with given figure numbers, if mentioned in the text).
- Chemical structures, computer generated! or (hand drawn in black ink acceptable).
- Theory
- Experimental Method
  Do not write using "cookbook" sentences. Describe what you did using your own words.

Results:
Are the calculations easy to follow? Are statistics used properly?
Is error treatment discussed? Do you use the correct number of significant figures? (10 pts)
Accuracy (30 pts)
Precision (10 pts)

Questions Must be answered! (10 pts)

Total (100 pts)

NOTE: The above grading scheme is subject to change based on the type of information being obtained in the experiment. For instance, if there are not questions to be answered then those points would not be included and the grade would be computed on a percentage basis of the total amount of points possible for the respective report.