Course Syllabus
Chemistry 443L-020
Instrumental Analysis Lab

Course Description:  Spectrochemical and electrochemical methods of analysis.

Number of Credit Hours:  0 semester hours - 3 hours lab per week

Course Prerequisites and Corequisites:  Prerequisite: 231 and 337. Required lab fee.

Program Learning Outcomes:
1. The student will integrate knowledge with critical thinking to solve problems.
2. The student will perform qualitative/quantitative chemical analyses/syntheses using modern instrumentation.
3. The student will articulate scientific information through written communication.

General Education Core Curriculum Objectives:  There are no specific general education core curriculum objectives in this course. This course is not a general education core curriculum course.

Course Objective:  Laboratory techniques will be demonstrated that are applied to instrumental analysis of chemical samples and solution chemistry. The basics of statistics related to analytical chemistry will be demonstrated by the students. When possible the analyses will be related to practical problem solving of contemporary, historical, technological, and societal issues.

Student Learning Outcomes:  The student is expected to demonstrate and apply the following concepts to problem solving:
- The calculations involved in the preparation of solutions using solid and liquid solutes.
- The basics of preparing standards, calibration curves, and validation of experimental analysis.
- Beer’s Law and how it is applied to instrumental analysis, involving atomic absorption spectroscopy, UV-Visible spectroscopy, and infrared spectroscopy.
- Principles of electrogravimetric analysis that involves special sample preparation using fuming solutions with sulfuric acid.
- The students should recognize the basic principles of potentiometry.
CHE 443L -020 F2012

Lab Syllabus
Fall 2012
CHE 443L-020
Instrumental Analysis Laboratory

Instructor: Dr. Darrell R. Fry
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Office: NM-120
Phone: 936-468-1406
Office Hours: MWF 9-10; W8-9; TR 8:00-9:30; R 2-5
Lab times: F 1:00-3:50 p.m. in Rm. C-306

TEXTS AND MATERIALS:
- A scientific calculator; lab book with carbon pages (may use an old lab book) & flash drive

SUPPLEMENTAL TEXT:

GRADING: Grades are based upon performance. A single letter grade will be assigned for the lab and the lecture. Seventy five percent of the combined grade comes from the course while twenty five percent comes from the laboratory. The table below describes the percentage of each assessment category towards the combined lab/lecture grade—following this is a description of the lab categories.

<table>
<thead>
<tr>
<th>Assessment</th>
<th>Percentage of combined grade</th>
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</thead>
<tbody>
<tr>
<td>Exam I on 9/20</td>
<td>10</td>
</tr>
<tr>
<td>Exam II on 10/11</td>
<td>10</td>
</tr>
<tr>
<td>Exam III on 10/25</td>
<td>10</td>
</tr>
<tr>
<td>Exam IV on 11/15</td>
<td>10</td>
</tr>
<tr>
<td>Quizzes/homework (~weekly)</td>
<td>10</td>
</tr>
<tr>
<td>Final Exam 12/12 @ 10:30</td>
<td>25</td>
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<tr>
<td>(Comprehensive)</td>
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</tr>
<tr>
<td>Weekly prelab material</td>
<td>5</td>
</tr>
<tr>
<td>Results</td>
<td>5</td>
</tr>
<tr>
<td>Written Reports</td>
<td>10</td>
</tr>
<tr>
<td>Other Reports</td>
<td>5</td>
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</tbody>
</table>

Grading scale: Recall that a single letter grade will be given for the lab and lecture. The line below gives the cutoffs for the various grades.
A: 100-90% B: 89-80% C: 79-70% D: 69-60% F: below 60%

Weekly Prelab material: 10 points per prelab. You will need to prepare for each lab before lab starts. Preparation includes the following
1) Knowing what experiment you will be performing and with whom.
2) Reading and understanding the procedure for the day.
3) Writing a short purpose statement in your lab notebook.
4) Clearly outlining the procedure in your lab notebook prior to lab.
5) Answering any prelab questions that are required.
6) Showing up to lab on time in the appropriate clothing (closed toe shoes, long pants, goggles, etc…)

**Results:** 15 points per experiment. Results matter! Since you will be working in groups, you will presumably have the same results as everyone in your group. Moreover, you will also be depending upon others in the group for a portion of your grade! Pay attention to the technique of others within your group—if you think they are doing it wrong, ask them! If a student is not is not participating, then the points on the results section will be lower than the others in the group.

**Written Reports:** 7 formal reports worth 30 points each are required. The formal reports are required for The Fourier Transform & Excel; NMR; GC/MS; IR; UV/VIS; AA and fluorimeter. “Other” reports are due for the Pogil activities, the statistics lab, and the calibration curve experiment. The formal reports are worth 30 point

**Guidelines for the Formal Laboratory Reports**
1) Formal reports are required for 7 experiments.
2) A lab report for each of these must be submitted; failure to do so will result in an F for the course. Lab reports are due one week from the date you finish performing the experiment. Late lab reports will be docked 2 point per day (including holidays and weekends).
3) The report should be of publication quality in content, writing and style of presentation. All reports must conform to the following guidelines
   • The report should have a cover page listing the title of the experiment, the name of the student, identities of other group members, the course number and date (first day of execution)
   • The report should contain an Abstract, Introduction, Experimental, Results and Discussion, and Conclusion sections
   • **Only typed reports report will be graded.** Use subscripts, italics and Greek symbols as appropriate. Do not use shorthand notation, computer notation or slang. For example, use $\alpha$ instead of alpha, $1.5 \times 10^{-5}$ not $1.5E-5$, $K_a$ not $Ka$, $x$ not $x^2$. Do not write special characters by hand. Learn to create the necessary symbols and notation using computer software. All equations must be generated with an equation editor (e.g. Microsoft equation).
   • All tables should contain a caption and should be numbered sequentially (do not use Roman numerals). Tables should be generated with an appropriate software (word, excel, etc).
   • All figures should contain a caption and should be numbered sequentially. Graphs should be generated with appropriate software.
   • Tables and figures may be included with text or may be placed on a separate page.
   • All sources should be cited using the format employed in the Analytical Chemistry Journal. Lab reports are due one week from the date you finish performing the experiment. Late lab reports will be docked 2 point per day (including holidays and weekends).
4) The grading scheme for lab reports will be as follows:
   A. Abstract – 3pts
      In approximately 250 words describe the purpose, analytical procedure, results and give some measure of the error associated with the experiment.
   B. Introduction – 2pts
      Write, in your own words, a brief introduction to the experiment, clearly stating the purpose of the experiment at the closing of the introduction.
   C. Experimental Section – 5pts
      Describe the instrument used. Digitally schematic the instrument and label the components. Reference the Lab Manuals and other for the Procedures.
   D. Results – 7 pts
      a) Raw data: present raw data in a suitable format. Be sure to title the tables properly.
b) Clearly list your results. Sample calculations and error analysis should be shown.
c) Include all plots and printouts. Spectra, chromatograms, etc. should be placed at the end of the report.

E. Discussion and Questions – 5pts
Discuss any results as required by the handout. Answer all questions listed in the handout. Rewrite every question using the numbering in the experiment handout.

F. Conclusion – 2pts
Comparisons to known values should always be made whenever possible. Estimation of errors should be discussed including possible sources of errors. Suggest any possible improvements in the experiment.

G. References – 3pts
Include references using the ACS format used in the journal Analytical Chemistry; i.e.: number all references consecutively in parentheses and include them at the end of your report in a section titled references.

H. Overall Quality – 3pts

GROUP WORK:
Students will work in groups to complete both the experiments required for the formal reports and in groups to complete some of the “other” reports. While roles may be assigned for the various groups students should treat others as peers. Working together requires everyone to work—not just a few people. Students who are not contributing will find a negative impact on their grade. Students should also realize that in a short period of time they may be expected to complete all of the tasks the group has been given independently—this is after all a senior level course.

Students will often work in groups of no more than 4 to perform the various experiments required for the formal reports; however, each student is required to complete a lab notebook and their own individual laboratory reports. Groups will be assigned by the faculty member and may be scrambled as the semester progresses. At the end of each lab period, the notebook pages for each student must be signed by the teaching assistant or instructor, and the duplicate (carbon copy) pages handed in. (Late submissions will not be accepted). Copies of graphs and printouts do not need to be submitted with the notebook pages, but must accompany the written report submitted by each student.

Students will work in groups of no more than 4 to complete the various POGIL activities. POGIL is an acronym for Process Oriented Guided Inquiry Learning. While roles may be assigned for the POGIL activities students should strive to treat each other as peers. Students not contributing to the POGIL activities will receive lower grades than everyone else.

The formats for the lab write-ups are described on this syllabus. Although the experiments are done in teams, all lab reports should be prepared and submitted individually. Copying and paraphrasing are not allowed, and will be treated as plagiarism, according to University policy. All written lab reports are due exactly one week following the lab period in which the experiment was completed, by 5:00 PM. Late lab reports will be penalize at a rate of 2 points per day it is late not to exceed 15 points.

SCOPE OF WORK: Adequate preparation, good delegation of tasks and a focus on the experiment being performed are necessary in order to perform the various experiments in the allotted time period. Adequate preparation is more than pre-reading the lab; it is carefully thinking through the procedure. As evidence of having accomplished this, often times students lab notebooks will be checked as they enter the laboratory. The laboratory notebook should contain a purpose statement and procedure with the necessary calculations complete prior to lab. As a recommendation, group members should meet briefly prior to lab to discuss how they will divide the duties. Sometimes work out of assigned class period is required. This will be compensated by
cancelling laboratory periods and having short labs. **Each student will be monitored to ensure proper rotation of work.**

**Guidelines for lab notebooks**
- Carbonless lab notebook; used appropriately.
  - Fill out partners. Fill out experiment number. Give a short description of your work in the appropriate blank. Signed before leaving, etc…
- Entries are written in ink.
- Table of contents up to date.
- Blank pages have a single diagonal line indicating that they are intentionally left out.
- Incorrect entries are crossed out with a single line.
- Written as experimental work proceeds (not the next day).
- Contains drawings of experimental apparatus, descriptions of how work was actually carried out (when changes are made in the lab manual procedure, you should indicate what you did, not what the lab manual said), and experimental observations.
- Includes results, equations and calculations.
- Includes explanations, interpretations, questions that arise, future plans.
- The notebook will be checked weekly. Students will tear out their duplicate pages and turn them in.

**LABORATORY POLICIES**

*Attendance Policy:* Attendance of laboratory is mandatory. A total of 2 unexcused absences may result in the student being dropped from the laboratory (and class!) with a grade of "F". Excused absences are University Recognized excused absences and illness/emergencies as deemed (and often verified) by the faculty member. On days where a group of instruments will be explained, but no data will be collected, attendance is still mandatory! A single make-up will be given towards the end of the semester.

*Preparation Policy*
- Success and enjoyment in the lab can come only from a thorough understanding of what you are doing. This can be achieved only with proper preparation before performing the experiment. Read the description of the experiment in the lab handout before you do the scheduled experiment.
- You may be asked to show your copy of the purpose and execution plan before you are allowed to perform your experiment.
- If it is determined that you have not read the materials before the lab, you will be asked to leave and a 5% deduction will be applied to your grade. Arrange at the lab on time. You will need most of the time allocated to complete each experiment, as you must first familiarize yourself with the instrument and prepare solutions. If you are more than 30 minutes late, you will not be permitted to start the experiment. Students who are *habitually* late (i.e. more than twice consecutively, or more than three times during the semester) without good reason will similarly be refused permission to perform experiments or submit pre-lab questions and reports.

*Lab Safety Policies*
1. Know the location and use of the fire extinguishers, eye wash/shower and first aid kit.
2. See your instructor if you are not familiar with the use of the safety equipment.
3. Wear glasses or safety goggles at all times in the laboratory.
4. Open-toed shoes of any type are strictly forbidden; SOCKS are required!!
5. No smoking or eating is allowed in the laboratory.
6. Nail polish should not be worn during the GC or FT-IR laboratories, as you will be working with organic solvents.
7. Gloves should be worn when handling strong caustics (acids, bases) and organic solvents. Shorts or short skirts should not be worn during in the labs.
8. Always keep your work areas and surroundings clean including the balance tables.
9. Be careful when you handle the electric instruments. If you are unfamiliar with the equipment, ask the instructor for help before you attempt to use them.
10. Pour organic and toxic inorganic residues into the appropriate waste bottles in the hood.
11. If you do not follow these safety guidelines, you will be asked to leave the laboratory, and you will receive no credit for that portion of the experiment.

**Responsible use of instrumentation and glassware:**
The instrumentation you will be using in laboratory is used both for teaching and research throughout the year. You can therefore appreciate the need to maintain it in good working order, and to keep the area around the instrument clean. It is important for you to be prepared for the experiments you perform, in order to maximize your learning experience and minimize the possibility of breakage. Your preparation should consist of: (1) reading the background chapter in Skoog, Holler & Nieman (see syllabus); (2) reviewing relevant lecture notes, and (3) preparing your lab notebook before the lab period. [You may also wish to consult materials from the library, particularly for your written reports.] Please respect the equipment, and recognize that breakage can be costly (money and down time). The instructor is available to help you if you are uncertain of how to proceed. We recognize that occasionally, things do break down. It is your responsibility to report any problems to the instructor immediately.

Likewise, the glassware you will be using in laboratory is also used for teaching and research throughout the year. Nothing is more frustrating than having to find and then clean glassware prior to starting an experiment. For this reason, all glassware will be cleaned prior to leaving laboratory and placed back into its labeled drawer. The instructor will indicate which lab drawers students are allowed to use. Several experiments will be conducted outside of CHE 306. DO NOT MEDDLE in drawers of other labs!! Again, the instructor will indicate which drawers contain which equipment/glassware prior to the experiment.

**Clean-up and hazardous waste:**
All used glassware should be properly cleaned and returned to its original location at the end of the lab period or after completing the experiment. Final rinsing should be with distilled water. Spills should be cleaned up promptly. The area around each instrument must be left clean at the end of the day, and will be inspected by the teaching assistant or instructor before signing your lab notebook at the end of the day. Attendance on the last lab day is required. At this time, any remaining glassware and samples will be dealt with, and the TA will approve your check-out from the lab. Many of the samples, including aqueous unknowns, may be considered hazardous wastes. Please follow the guidelines provided by instructor for proper disposal in marked containers. Failure to follow proper waste disposal procedures results in substantial costs to the department, ca. $100 per 5 gal. bottle that must be re-separated or re-analyzed. Anyone found improperly disposing of wastes will be charged these costs or be sent out of the lab altogether.

**MAKE-UP POLICY:** no labs will be made up. If a student has a documented excludes absence, then they may use other people’s results (reference). The lowest other lab report will be dropped.

**ACADEMIC INTEGRITY (A-9.1):**
Academic integrity is a responsibility of all university faculty and students. Faculty members promote academic integrity in multiple ways including instruction on the components of academic honesty, as well as abiding by university policy on penalties for cheating and plagiarism.
**Definition of Academic Dishonesty**

Academic dishonesty includes both cheating and plagiarism. Cheating includes but is not limited to (1) using or attempting to use unauthorized materials to aid in achieving a better grade on a component of a class; (2) the falsification or invention of any information, including citations, on an assigned exercise; and/or (3) helping or attempting to help another in an act of cheating or plagiarism. Plagiarism is presenting the words or ideas of another person as if they were your own. Examples of plagiarism are (1) submitting an assignment as if it were one's own work when, in fact, it is at least partly the work of another; (2) submitting a work that has been purchased or otherwise obtained from an Internet source or another source; and (3) incorporating the words or ideas of an author into one's paper without giving the author due credit.

Please read the complete policy at [http://www.sfasu.edu/policies/academic_integrity.asp](http://www.sfasu.edu/policies/academic_integrity.asp)

Any student found cheating will be subject to the penalties as stated in the Student Code of Conduct handbook; including but not limited to a score of zero on exam, expulsion from the class or expulsion from the University.

**Withheld Grades Semester Grades Policy (A-54):**

Ordinarily, at the discretion of the instructor of record and with the approval of the academic chair/director, a grade of WH will be assigned only if the student cannot complete the course work because of unavoidable circumstances. Students must complete the work within one calendar year from the end of the semester in which they receive a WH, or the grade automatically becomes an F. If students register for the same course in future terms the WH will automatically become an F and will be counted as a repeated course for the purpose of computing the grade point average.

The circumstances precipitating the request must have occurred after the last day in which a student could withdraw from a course. Students requesting a WH must be passing the course with a minimum projected grade of C.

**Students with Disabilities:**

To obtain disability related accommodations, alternate formats and/or auxiliary aids, students with disabilities must contact the Office of Disability Services (ODS), Human Services Building, and Room 325, 468-3004 / 468-1004 (TDD) as early as possible in the semester. Once verified, ODS will notify the course instructor and outline the accommodation and/or auxiliary aids to be provided. Failure to request services in a timely manner may delay your accommodations. For additional information, go to [http://www.sfasu.edu/disabilityservices/](http://www.sfasu.edu/disabilityservices/).

**Classroom Behavior Policy:** To ensure a classroom environment conducive to learning, any forms of classroom disruptions will not be tolerated (examples but not limited to – talking, use of cell phones/beepers, sleeping, reading other material, eating/drinking). Students who violate these rules will be asked to leave. Repeat offenders will be subject to disciplinary action in accordance with University policies as described in the Code of Student Conduct. All necessary/required parts of the day’s experiment **must be done prior to class.**
## Tenative Schedule

<table>
<thead>
<tr>
<th>Week</th>
<th>Date</th>
<th>Experiment Description</th>
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<tbody>
<tr>
<td>1</td>
<td>8/31</td>
<td>Safety (POGIL)</td>
</tr>
<tr>
<td>2</td>
<td>9/7</td>
<td>Statistics (POGIL)</td>
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<tr>
<td>3</td>
<td>9/14</td>
<td>Fourier Transform &amp; Excel</td>
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<tr>
<td>4</td>
<td>9/21</td>
<td>Group 1 Introduction: NMR, GC/MS and IR (POGIL)</td>
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<tr>
<td>5</td>
<td>9/28</td>
<td>Rotate through NMR, GC/MS and IR</td>
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<td>6</td>
<td>10/5</td>
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<td>7</td>
<td>10/12</td>
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<tr>
<td>8</td>
<td>10/19</td>
<td>Group 2 Introduction: UV/VIS, AA, Calibration Curve (POGIL)</td>
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<td>9</td>
<td>10/26</td>
<td>Rotate through UV/VIS, AA and Calibration Curve</td>
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<td>10</td>
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<td>11</td>
<td>11/9</td>
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<tr>
<td>12</td>
<td>11/16</td>
<td>Fluorimeter/POGIL</td>
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<tr>
<td>13</td>
<td>Thanksgiving</td>
<td>No Lab</td>
</tr>
<tr>
<td>14</td>
<td>11/13</td>
<td>Fluorimeter/POGIL</td>
</tr>
<tr>
<td>15</td>
<td>11/7</td>
<td>All lab reports to be graded are due</td>
</tr>
<tr>
<td>16</td>
<td>Finals week</td>
<td>No laboratory</td>
</tr>
</tbody>
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