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.
Class Syllabus
Fall 2017
CHE 443L-020
Instrumental Analysis Laboratory

Instructor: Dr. Kefa K. Onchoke
Department: Chemistry
e-mail: onchokek@sfasu.edu Please use this e-mail to assure timely response from instructor.
Office: NM-118
Phone: 936-468-2386
Office Hours: M 10-12; T 11-1; W 4 – 5 p.m; R 4 – 5 p.m.
Lecture times: F 1.00-3.50 p.m. in Rm. C-106

TEXTS AND MATERIALS:
2. Other articles and spectra handouts will be distributed in the course as required reading.


GROUP WORK:
Students will work in groups of no more than 3 and no more than two groups per lab section. The group will be responsible for assigning work associated with the making of standards and the operation of each piece of equipment.

All experiments will be done by students working in pairs. Each student is required to keep a lab notebook, complete with data and descriptive results. At the end of each lab period, the notebook pages 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.

The formats for the lab write-ups are described on this syllabus (page 5). 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 penalized at a rate of 20% per day (including weekends).

SCOPE OF WORK: Students must pre-read the lab. Sometimes work out of assigned class period is required. This will be compensated by cancelling lecture periods. Each student will be monitored to ensure proper rotation of work.
COURSE CALENDER & CONTENT:
Laboratory Operation

1. You will do the experiments in teams of two or three individuals.

2. You must read the manual and any relevant materials from the textbook for the scheduled experiment prior to the lab period assigned to you (see lab policy). It can be difficult to conceptualize an instrument you have not used before; however, you must make an effort to understand as much as possible from the reading of the handouts. If you can, you may visit the experimental station before hand to help clarify some of your readings, especially on the instruments’ components and operation. I strongly encourage you to 'visit the instrument' you will use while you are reading instructions.

3. At the beginning of each experiment, the instructor will show you how to operate the equipment.

4. At the end of your experiment dispose of all the solutions you prepared yourself and clean all the flasks. If you find the flasks dirty or containing solutions, report it to the instructor.

5. Some solutions will be shared. Please use proper laboratory techniques to help yourself from reagent containers. Failure to do so will cost you some points if the instructor becomes aware of it.

LABORATORY POLICY

1. 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.

2. You must hand in a copy of the introduction, calculations required and a brief execution plan before you are allowed to perform your experiment.

3. 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.

4. 10% of your score for each experiment is based on your preparation and laboratory technique as determined by the instructor.

5. Arrive 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.
Lab Safety

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.

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 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.

If you have an accident, notify the instructor immediately.

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.
Guidelines for Laboratory notebooks

- Bound.

- Entries are written in ink.

- Two-three pages at the front are dedicated to a table of content.

- Each page is dated and numbered.

- 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.

Laboratory Report

1. Formal reports are required for all experiments.

2. 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, the course number and date (first day of execution)

   - The report should contain an Abstract, Introduction, Experimental, Results, 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, $x^2$ 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.

3. The grading scheme for lab reports will be as follows:

I. Abstract - 5%

II. Introduction - 5%
Write, in your own words, a brief introduction to the experiment, clearly stating the purpose of the experiment at the closing of the introduction.

III. Experimental Section - 5%
Describe the main experimental setup. Roughly sketch the setup and label the components. Reference the Lab Manuals and other for the Procedures.

IV. Results - 40%

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.

V. Discussion and Questions - 30%
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.

VI. Conclusion – 5%
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.
VII. References - 5%.

(i) 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. At least 10 journal articles must be cited. The journal articles must be within ±10 years from September 1, 2017. UNLESS THE JOURNAL ARTICLE IS A MAJOR ARTICLE IN THE FIELD

(ii) No university websites are to be cited.

(iii) No websites or Wikipedia citations.

4. Lab reports are due one week from the date you finish performing the experiment.

5. To get feedback from the instructor on your reports, the lab report will be graded within one week after you hand it in and will be handed back to you during the following lab period.

6. You can discuss with the instructor the graded report in the lab and she/he may provide you with some valuable comments so that you can improve your next lab report. **You must hand back the graded report to the instructor at the end of the lab period.**

What you turn in:

a) The typed report
b) **Work Distribution Form**, with division of labor signed by each student
c) Carbon copy from page of lab book from each student.
d) Individual data as relevant.

When you turn it in:

Next lab
 Provisional grade given
 Edited and redone material with the original submission containing faculty commentary must be re-turned in 1 week after faculty edits. No resubmission is encouraged.

**Responsible use of instrumentation:**
The instrumentation you will be using in CHE 443L 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.

**During the lab:** The lab notebook should be your constant companion. Follow good laboratory notebook practice, just as you would be expected to in industry or at the NIH. All entries should be complete and legible. *Nothing* should be written on scraps of paper; everything is entered in the lab notebook. Record procedures (e.g., how serial dilutions were made, or how solutions of solids were prepared), any qualitative observations (e.g., appearance of solutions, difficulties encountered during sample preparation, etc.) as well as quantitative observations. Spectra or other information that are printed or plotted separately should be labeled clearly, titled, and marked with your initials and lab notebook page number. You should record the creation of such output in your notebook.

**At the end of the lab:** The instructor or teaching assistant will sign each page of your lab notebook for the day (do NOT tear out the sheets before they are signed!), and collect the carbon-copy pages. [No credit will be given for notebook pages that are submitted unsigned or late.] You do not need to submit copies of spectra or chart outputs with those pages, but the existence of such data must be clearly indicated in your notebook entries. At that time, the teaching assistant will also check that the instrument and sample preparation areas are clean, before you leave for the day.

- Notebook grading: to help you in keeping a better notebook, your submitted pages will be graded and shown to you as the course proceeds. They will be kept on file until the end of the course. **All lab reports must be typed.** Term work written in pencil will not be accepted for remarking

**GRADING:** No make-up labs will be given for any reason.

**LIST OF EXPERIMENTS**

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<thead>
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<tbody>
<tr>
<td>1</td>
<td>Simulation expt (UV-vis)</td>
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<td>2</td>
<td>IR</td>
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<tr>
<td>3</td>
<td>Atomic Absorption (AA)</td>
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<td>4</td>
<td>UV-Vis</td>
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<td>5</td>
<td>Cyclic voltammetry</td>
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<td>6</td>
<td>Molecular fluorescence</td>
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<td>7</td>
<td>GC-MS</td>
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<td>8</td>
<td>Ion chromatograph</td>
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<tr>
<td>9</td>
<td>ICP-OES, ICP-MS</td>
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<tr>
<td>10</td>
<td>NMR (¹H and ¹³C)</td>
</tr>
<tr>
<td>11</td>
<td>NMR – 2D experiments</td>
</tr>
</tbody>
</table>
**1. One more experiment - HPLC- is likely to be added. You will be notified in time and informed on the scheduling changes.

2. Groups (2 individuals) will be assigned to overseeing the proper maintenance of given instruments during the semester. At the end of the semester 5-10 bonus points will be awarded for perfectly overseeing the instrument.

**COURSE SCHEDULE**

Note: A Course schedule will be handed in class after the first week of class. The schedule is tentatively sketched out below.

CHE 443L - Lab schedule

Fall 2017 (September/October schedule)

<table>
<thead>
<tr>
<th>Group/Dates</th>
<th>Lab students Group 1</th>
<th>Lab students Group 2</th>
<th>Lab Group Group 3</th>
<th>Lab Group Group 4</th>
</tr>
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<tbody>
<tr>
<td></td>
<td>Forrester Franclemont Herbert</td>
<td>McClinton Nguyen Orellana</td>
<td>Perez Rackley Shah Lillie</td>
<td>Spurr Stager Thompson</td>
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<tr>
<td>Sept 1</td>
<td>syllabus</td>
<td>syllabus</td>
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<td>syllabus</td>
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<tr>
<td>8-Sept</td>
<td>Data treatment/ simulation</td>
<td>Data treatment/ simulation</td>
<td>Data treatment/</td>
<td>Data treatment/</td>
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<tr>
<td>22-Sept</td>
<td>UV/Vis</td>
<td>IR</td>
<td>GC-MS</td>
<td>IC</td>
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<tr>
<td>29-Sept</td>
<td>IC</td>
<td>UV/vis</td>
<td>IR</td>
<td>GCMS</td>
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<tr>
<td>6-Oct</td>
<td>GCMS</td>
<td>IC</td>
<td>UV/vis</td>
<td>IR</td>
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<tr>
<td>13-Oct</td>
<td>IR</td>
<td>GC-MS</td>
<td>IC</td>
<td>UV/vis</td>
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<td>27-Oct</td>
<td>CV</td>
<td>AA</td>
<td>Fluorescence</td>
<td>ICP-MS</td>
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<td>3-Nov</td>
<td>ICP-MS</td>
<td>CV</td>
<td>AA</td>
<td>Fluorescence</td>
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<tr>
<td>10-Nov</td>
<td>Fluorescence</td>
<td>ICP-MS</td>
<td>CV</td>
<td>AA</td>
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<tr>
<td>17-Nov</td>
<td>AA</td>
<td>Fluorescence</td>
<td>ICP-MS</td>
<td>CV</td>
</tr>
<tr>
<td>24-Nov</td>
<td>NMR (13C)</td>
<td>NMR (13C)</td>
<td>NMR (15C)</td>
<td>NMR (13C)</td>
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<tr>
<td>Dec. 1</td>
<td>NMR (1C)</td>
<td>NMR (1H)</td>
<td>NMR (1H)</td>
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**NOTE: THIS IS A TENTATIVE SCHEDULE. THE COMPLETE SCHEDULE OF EXPERIMENTS WILL BE PROVIDED ON WEDNESDAY, SEPTEMBER 4/2013.**
Remarking of Returned Term Work:

- Errors in marks addition on returned term work should be brought to the attention of the course instructor within two weeks or the end of the December examinations period, whichever comes sooner.
- Graded lab reports may be allowed to resubmit that report, at the discretion of the course instructor; a resubmitted report must be accompanied by the original report.

Lateness to the Laboratory:

- Students are reminded that the laboratory classes are mandatory.
- Labs start at 1:00 pm, not 1:10 p.m; a progressive lateness penalty applies (10 pts each time) to students who come in late.
- Students who are more than 30 minutes late for a regularly scheduled laboratory class without good reason will not be allowed to perform that experiment, nor submit their pre-lab questions or a report for grading (i.e. will receive a zero mark for the entire 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.

Missed Quizzes, and Labs:

- Students who miss a lab session for a valid reason (such as a medical emergency) should contact the course instructor in writing as soon as possible; students may be offered either an opportunity to make up the missed work or consideration for the lost marks, depending on circumstances.

Final Exam – There will be a comprehensive final given during class on Dec. 6/2017. The Final exam is worth 100 pts. All experiments done during the semester will be tested.

MAKE-UP POLICY: There will be no make-up exams, quizzes, or labs.

ATTENDANCE POLICY: Attendance of class is mandatory. A total of two unexcused absences will result in the student being dropped from the class with a grade of "F".

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
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/.

SEMESTER WITHDRAWALS: Last day to withdraw from the course without obtaining WP or WF grade is October 23.

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.