Instrumental Analysis Lab  
Chem 426  
2 Credits, M

Course Date:  Tues. Aug., 20, 2019 through Thurs., Dec., 5, 2019  
Location:  Fulmer 219  
Meeting day: Tues and Thurs  
Meeting Time:  1:25 to 4:15 PM  
Prerequisite(s): Chem 425 with a C or better

Instructor Information  
Prof. Jeremy Lessmann  
Email:  jlessman@wsu.edu  
Office: Fulmer 311  
Office Hours: By Appointment  
Phone: 509-335-2098  
TA:  Adam Huntley, adam.huntley@wsu.edu and office hours TBA

Student Learning Outcomes: (Chemistry Dept. Outcomes in ())  
At the end of this course student will be able to:  
1. Explain the principles behind and operate the most commonly encountered laboratory instrumentation for chemical analysis. (2,5)  
2. Determine the most useful Figures of Merit for instrumentation. (2,3)  
3. Design experiments and perform the 3 common methods of quantitative analysis using instruments. (3,5)  
4. Apply appropriate statistical analysis of data. (3)  
5. Communicate experimental results in the proper written formats for Chemistry. (4)

Assessment of Learning Outcomes:  
Assessments of the above learning outcomes are provided for in the student prepared laboratory reports. Learning Goal 5 in particular will be assessed in the two formal reports.

Lab Schedule (Depending on what topics we need to review this schedule may be revised on the fly)  
Tues Aug 20 Intro and start of Chromatography and Electrochem lectures.  
Thurs Aug 22-Thurs Sept 19 Chromatography and Electrochem Lectures  
Tues Sept 24: Check in and Experiments begin: Students will be rotating through 6 experiments over the rest of the semester  
Each experiment should take 3 lab periods. Specific schedule will be discussed on the first day.  
Tues Nov 25-29 No Lab Thanksgiving Break  
Thurs Dec 5 Cleanup and checkout day
General Lab Experiments and Lab Report Requirements:

You will be performing 6 labs over the course of the semester. Students will work in pairs on the instruments but each student will have their own unknown to analyze. Both students are expected to learn how the instruments work equally and how you do is part of your technique grade. The following figures of merit must be determined in addition to any other requested analysis for a given experiment:

**Figures of Merit Checklist:**
1. Calibration plot or standard addition plot, or internal standard data
2. Equation of calibration or standard addition fit with standard deviations of slope and intercept or statistical data for internal standard analysis
3. Table of true concentrations (standards and pseudo-unknown), estimated concentrations with standard deviations (from equations not repeat trials), and RSD’s.
4. Sensitivity
5. Detection limit
6. Spectrum, chromatogram, voltammogram or whatever is appropriate for the instrument, printed from Excel and in proper format for publication in the ACS Journal *Analytical Chemistry* (Consult the Author Guidelines under Submission & Review ([http://pubs.acs.org/journal/ancham](http://pubs.acs.org/journal/ancham))

**Experiment List (Students will choose 6 of the 8)**
1) Basic Radiochemical Measurements and environmental radioactivity (Geiger tubes and Gamma spectroscopy)
2) GC-MS analysis of aromatic compounds in gasoline
3) HPLC analysis of caffeine in coffee and tea
4) Flame-AA and ICP analysis of minerals in apples
5) Spectrofluorometric analysis of quinine
6) Voltammetric methods of analysis
7) UV-Vis determination of equilibrium constant for a reaction

**Standard lab reports:** Follow the information on what constitutes a good lab report found on the course’s Blackboard page. Reports are due three lab periods after you finish the experiment.

**Formal Lab reports:** For the 2nd and 5th experiments you perform you will be submitting your report as if it were a manuscript for publication following the American Chemical Society’s journal requirements for *Analytical Chemistry*. Information on what constitutes proper formatting and style (esp. reference format) can be found on the information for authors by following the ACS url above. The report should be at least 8 pages including figures and references. These two reports will be returned to you, and you will have 1 week from the return date to make corrections and return them for reevaluation. This is much like the peer review process we use to publish research. The
revision will be graded an additional 25 points which are partially based on how well you incorporated the reviewer’s suggestions. This means the total points available for a formal report are 75.

**Grading Policy:** You must turn in a report for every experiment to be eligible to pass the course.

<table>
<thead>
<tr>
<th>Assignment</th>
<th>Possible Points</th>
<th>Percent of Grade</th>
</tr>
</thead>
<tbody>
<tr>
<td>Experiment reports (5@ 50)</td>
<td>250</td>
<td>50 %</td>
</tr>
<tr>
<td>Formal reports (2@ 75)</td>
<td>150</td>
<td>30 %</td>
</tr>
<tr>
<td>Lecture Problem sets (2@25)</td>
<td>50</td>
<td>10 %</td>
</tr>
<tr>
<td>Lab Notebook and Recordkeeping 25</td>
<td></td>
<td>5 %</td>
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<tr>
<td>TA and Instructor Assessment</td>
<td></td>
<td>5 %</td>
</tr>
<tr>
<td>Total points possible</td>
<td>500</td>
<td>100%</td>
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Standard rounding rules will be used to calculate letter grades.

**Grade Cutoffs:**
- Above 459 points A
- 459-435 A-
- 434-420 B+
- 419-400 B
- 399-385 B-
- 384-370 C+
- 369-350 C
- 349-335 C-
- 334-320 D+
- 319-300 D
- Below 300 points F

**Academic Integrity:** I encourage you to work with classmates on assignments and you will be working with partners for some experiments. However, each student must turn in original work. No copying or sharing of spreadsheets will be accepted. Students who violate WSU’s Standards of Conduct for Students may receive an F as a final grade in this course, will not have the option to withdraw from the course and will be reported to the Office of Student Conduct. Cheating is defined in the Standards for Student Conduct WAC 504-26-010 (3). It is strongly suggested that you read and understand these definitions.

**Students with Disabilities:** Reasonable accommodations are available for students with a documented disability. If you have a disability and need accommodations to fully participate in this class, please either visit or call the Access Center (Washington Building 217; 509-335-3417) to schedule an appointment with an Access Advisor. All accommodations MUST be approved through the Access Center. For more information contact a Disability Specialist at http://accesscenter.wsu.edu, or Access.Center@wsu.edu
Classroom Safety Information: Classroom and campus safety are of paramount importance at Washington State University, and are the shared responsibility of the entire campus population. WSU urges students to follow the “Alert, Assess, Act,” protocol for all types of emergencies and the “Run, Hide, Fight” response for an active shooter incident. Remain ALERT (through direct observation or emergency notification), ASSESS your specific situation, and ACT in the most appropriate way to assure your own safety (and the safety of others if you are able).

Please sign up for emergency alerts on your account at MyWSU. For more information on this subject, campus safety, and related topics, please view the FBI’s Run, Hide, Fight video and visit the WSU safety portal.
General Rubric for All Lab reports: (This will be modified to meet specific experiment circumstances)

<table>
<thead>
<tr>
<th>Category</th>
<th>Possible Points</th>
<th>Points Earned</th>
</tr>
</thead>
<tbody>
<tr>
<td>1) Grammar, proper references, etc</td>
<td>5</td>
<td></td>
</tr>
<tr>
<td>2) Intro – Explain instrumentation</td>
<td>10</td>
<td></td>
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<tr>
<td>3) Experimental section</td>
<td>5</td>
<td></td>
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<tr>
<td>3) Results/Discussion/Data – Print graphs etc including Figures of Merit from syllabus</td>
<td>20</td>
<td></td>
</tr>
<tr>
<td>4) Questions answered from lab/Conclusions</td>
<td>5</td>
<td></td>
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</tbody>
</table>

Total: 50 pts

Additional Rubric for Formal Reports

<table>
<thead>
<tr>
<th>Category</th>
<th>Possible Points</th>
<th>Point Earned</th>
</tr>
</thead>
<tbody>
<tr>
<td>1) Grammar revision, proper tense, etc</td>
<td>10</td>
<td></td>
</tr>
<tr>
<td>2) Proper Abstract</td>
<td>5</td>
<td></td>
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<tr>
<td>3) Figures in proper format for journal</td>
<td>5</td>
<td></td>
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<tr>
<td>4) Proper section heading</td>
<td>2</td>
<td></td>
</tr>
<tr>
<td>5) References in proper format</td>
<td>3</td>
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Total: 25 points
For an AWESOME WONDERFUL lab report!

**Grammar/Format:** *Double space, 12pt font, Times New Roman.* Please re-read your lab and make sure that your sentences make sense. Make sure that your format is logical and follows a traditional paper: (Abstract) Intro, Experimental, Results/description, conclusion etc. Your figures/graphs/tables should all be imbedded into the paper not attached as "extraneous information". Don't forget to superscript and subscript and use units and symbols! $10^{-3}$ M LaCbor $1.45 \pm 0.03$ MHzSO4 (hint: word has shortcuts for all these things, learn how to use them)

**Intro:** Explain the instrumentation you are using and give a little background on the lab. What does the instrument read? What information are you receiving from the instrument? What are you doing in the lab? Why is it important or fun to do this? Think of me as a sophomore with basic chemistry background, but no instrumentation background.

**Experimental:** Explain what you did and how you did it. If you performed calculations, what general equations did you use? Yes, you are following an established protocol and you should cite it, but remember you probably made changes to the protocol and those should be described here.

**Results/Discussion:** Your data should go in this section and you should be talking about it. Remember the rules for figures of merit—label everything so that I know what I am looking at. What should it tell me? Please don't use connecting lines on scatter plots, instead add a linear trend line with the equation and $R^2$ value displayed. Don't forget your sigfig rules: 1.4567 ± 0.025436 M becomes 1.45 ± 0.03 M. If your data is not in a format for you to imbed in your report (i.e. papers printed directly from the instrument) label them in an appendix and reference that in your lab report (see appendix table 1 for...).

**Conclusions:** Each lab has a series of questions throughout the lab and those questions should be answered in this section in paragraph format in a logical order. This shouldn't look like a general chemistry lab report with all the questions laid out. Explain what you did and answer the question that is associated with it. Finally, have a paragraph at the end with a summary of what you did in the lab from beginning to end and what results you got from it.

**Reminders:** If you look something up add a bibliography and cite your sources. If you didn't finish a portion of the lab mention it and talk about why. Your lab is supposed to tell a story. Pretend I wasn't sitting in lab—what did you do? I should be able to make a reasonable attempt to repeat the lab from what you wrote and get similar results.

If you have questions email me or make arrangements with me for office hours.