Analytical Instrumental Laboratory
CHEM 314; Spring 2022

Course Name: CHEM 314, 3 credits
Prerequisites: CHEM 212
Location: Reichardt 165 and Zoom (lecture); Reichardt 245 (lab)
Meeting Time: M 9:15-10:15am (lecture), MW 2:15-5:15pm (lab)
Final: 1-3 p.m., Friday, April 29 (based on lab meeting time)
Instructor: Dr. Jingqiu Mao
Office: Reichardt 188
Phone: 907-474-7118
Email: jmao2 at alaska.edu
Office Hours: By appointment

Recommended Materials:
Skoog, Holler and Crouch, **Principles of Instrumental Analysis**
Harris, **Quantitative Chemical Analysis**.
**** Current editions of textbooks on reserve in Rasmussen Library for 2 hr in-library use****

Course Description: A laboratory course focusing on the acquisition and interpretation of chromatographic and spectroscopic data for quantitative chemical measurements. Students will learn effective experimental planning and execution, critical evaluation of experimental data and written communication in the context of the chemical sciences. Much of this course is student-directed and project based. Students are expected to carefully prepare, plan, and execute experiments with minimal instructor input. CHEM 314 builds on previous experience with analytical chemistry (eg CHEM 212).

Instructional Methods: This class is based on characterizing several products given to each student. Throughout the semester, students will use a variety of instrumentation to characterize their products both individually and in groups. In lecture, students will learn to correctly apply, diagram, and troubleshoot instruments. They will apply this knowledge in lab by designing experiments and learning to use instruments to characterize their product. After each experiment is completed, written reports will be submitted for TA and instructor review.

Course Goals: Students will learn to design an experiment, select appropriate instrumentation, research and apply laboratory procedures, carry out experiments, troubleshoot instruments, analyze data, and write it all up in a scientific report.

Student Learning Outcomes:
- Students will be able to diagram spectroscopic and chromatographic instrumentation and select the appropriate instrument for a particular problem.
- Students will be able to research and apply instrumental methods for characterizing materials. Then perform the experiment and interpret the results.
- Students will compose a scientific report in the ACS style to justify and document experiments, interpret results, and draw conclusions.

Available Instrumentation: Flame AA, ICP-MS, FT-IR, UV-VIS, GC, GC-MS, HPLC, NMR.

Course Policies:
Continued attendance to class indicates each student agrees to the policies set forth in this syllabus.

Behavior and Collaboration- Students are expected to conduct themselves professionally at all times. Disrespect of the classroom learning environment, instructors, and fellow students is not tolerated! Collaboration and working in small groups is a key component of classroom and lab time.

Attendance, Tardiness, and Late Work- Students are expected to attend class and not compromise the experience of other students. Makeup labs are not available for this course except for school-related travel. Work is not accepted late. This is to keep us all moving though the material efficiently.

Instructor-Initiated Withdrawals- Any time up to and including the final date to drop a course with a "W," the professor has the right to withdraw a student that "...has not participated substantially in the course."

Honor code and Academic integrity- Students are expected to conduct themselves in accordance with the UAF Honor code. The Chemistry Department policy states: *Any student caught cheating will be assigned a course grade of F. The students academic advisor will be notified of this failing grade and the student will not be allowed to drop the course.*

Disability Services- I will work with the Office of Disabilities Services (208 Whitaker Bldg, 474-5655) to provide reasonable accommodation to students with disabilities. It is the student's responsibility to make an appointment with me to discuss appropriate accommodations. A letter from disabilities services must be provided.

CHEM 314 Evaluation

There are **1000 total points available** in this class. Grades are assigned on the typical scale 1000-900 A, 899-800 B, 799-700 C, etc.

Assignment	Points
Literature review & presentation	100
Reports (7@100 each)	550
Lab notebook	50
Exams (2@100 each)	200
Final presentation	100
Total points graded	**1000**

Literature review & Presentation (100 pts)- You will give a presentation based on a paper you are provided by the instructor. The presentation is supposed to identify the major components in that paper (see full report format below).
Final Presentation (100 pts)- You will give a final presentation at the end of the semester, based on one of your favorite experiments you have done in this semester. You will present in a similar fashion to your first presentation, but based on your own work!
Exams (200 pts)- Midterm and Final Exams will count for 100 points each.
Lab Notebook (50 pts, individual)- Maintaining a tidy, individual lab notebook is essential in science and is required for this course. It is your permanent record of your progress through this course that should be readily interpretable by anyone who picks it up- even me. Before coming to lab for experimentation, each student must have a procedure, and data tables prepared. Lab notebooks will be checked periodically throughout the semester without advanced notice. **Students must use a dedicated, organized bound notebook with sections for each experiment. Instrument data may be printed out and taped into the notebook. Loose papers or disorganized work is not acceptable.**

Experiments (550 pts; individual & group)- Experiment requirements vary throughout the semester

Experiment	Report	Points
Experiment 1: Standard addition	Full report- individual	100 pts
Experiment 2: Flame AA	Annotated Figs and Results-individual	50 pts
Experiment 3: FT/IR	Full report- individual	100 pts
Experiment 4: ICP-MS	Annotated Figs and Results-individual	50 pts
Experiment 5: HPLC	Full report- individual	100 pts
Experiment 6: GC or GC/MS	Annotated Figs and Results-individual	50 pts
Experiment 7: NMR	Full report- individual	100 pts
Total		**550 pts**

Full reports include: Procedure, Annotated Figures, and Report
Procedure- Prior to performing an experiment, groups will write a detailed description of their plan in their lab notebook. It will detail how you intend to prepare and analyze your samples by including: a purpose, step by step instructions to a level of detail that another student could easily replicate your work, a list of all reagents and materials needed, a description of what you expect to find and/or how you will analyze the data, and include references to any outside sources consulted.

Analytical Instrumental Laboratory
CHEM 314; Spring 2022

1. **Title**- Exciting or boring, be descriptive
2. **Purpose**- what are you trying to analyze and why?
3. **Background**-
   - **a.** Give a brief overview of the procedure and what you are analyzing for. What are the likely compounds you are looking for? Make a figure of their structures.
   - **b.** What are the anticipated results? Include literature spectra, chromatograms, etc have you found as examples. If applicable, make a table of physical properties (lamda max, molar absorptivity, etc) that will be useful or help you distinguish between the possible compounds.
   - **c.** Suggest alternate procedures that might work if this doesn't.
4. **Safety-** Print MSDS for all compounds used in your procedure.
   - **a.** List hazards associated with your procedure- either chemical or procedural
   - **b.** What engineering controls and PPE will be used to mitigate hazards?
   - **c.** What are the most likely things to go wrong? How will you react if they happen?
5. **Sample prep**- How will you prepare your sample? Go from the product to getting your sample into the instrument.
6. **Instrument parameters**-What conditions/ parameters are you going to use to run the instrument?
7. **Data tables-** What information will you record during the course of the experiment? Be sure you **SPEND SOME TIME** thinking though your experiment! And leave space in case things change during the experiment.
8. **Data Analysis-** Plan for statistical analysis of data, estimation of error, propagation of error.
9. **What do you need?** If you want what you need to be available, be sure to ask for it ahead of time! List what materials you will need: solvents, standards, glassware, etc. State the expected results are (retention time, etc).
10. **References**- Where'd you get your information and inspiration?
11. **Appendices-** Attach MSDS and key references or methods if applicable.

<u>Annotated Figures</u>- Print out copies of your figures and tables on individual pages. In addition to figures and tables of data you have generated, think of other materials you will need to include in your report. Examples: summary tables, structures of possible and identified molecules, spectra or other reference materials from literature.

On the print-outs, add a title and descriptive caption to figures and tables. Record observations (to go into the results section of your report), interpretations (to go into the discussion), and a sentence conclusion. These will be checked off in class and provide a starting point for group discussion and very helpful in writing the lab report.

Figure or Table title?	
Observations	Interpretations
Conclusion	

<u>Full Report Format</u>- All reports will be **written in a consistent journal format in a professional tone!**

1. **Descriptive title**- "Report 3" won't cut it.
2. **Abstract**- (250 words max)
3. **Introduction-** (2-3 paragraphs) Give some background on your question. Feel free to recycle from the project definition, but make it concise and coherent.
4. **Instrument Diagram**- (1-3 paragraphs) Diagram the instruments used and explain how the measurements are made.
5. **Materials and Methods**- (1-3 paragraphs) This is a paired down version your procedure. Include sample preparation procedures and instrument parameters and conditions.

6. **Results**- (1-3 paragraphs) Follows directly from annotated figures and include at least a paragraph for each figure and table. Reference figures in the text.
7. **Discussion**- (1-3 manageable paragraphs) Follows directly from annotated figures.
8. **Conclusions**- (2-5 sentences) saying what you found and what that means.
9. **Acknowledgments**: Research projects are collaborative and substantial contributions have been made by mentors, peers, postdocs, faculty, etc. Any person that significantly contributed to your work and their specific contribution should be mentioned.
10. **References**- If you used anything other than your brains (websites, text books, another group's procedure, instrument instructions), cite these resources appropriately!
11. **Figures and tables**- Each figure and table is to be numbered and on it's own page with a figure title and caption. All measurements should have an error estimate and graphs should have error bars.

Additional Guidelines for Written Assignments

- Abbreviations are often necessary and should be introduced clearly when used the first time.
- Element names (Iron) are spelled out only if they are the first word in a sentence and when they are part of a name (iron oxides). Otherwise, the symbol is used (Fe).
- Succinct description and clear reference in text to all figures and tables in the text.
- Do not duplicate data between the text and figures or tables
- Use SI units or the standard units in the field for all laboratory data. Appropriate formatting is used to indicate units. For example, mg kg^{-1} is correct, while mg/kg or ppm is not. Pay attention to these details in the literature you are reviewing.
- Use bold font to indicate references to figures, tables, and equations in the text. This helps during the proofreading process. Reference to **Figure 1** in the middle of a sentence and at the end of the sentence (**Fig. 1**). The word is always spelled out in entirety (**Table 1**).
- All figures and tables must have a descriptive caption clearly explaining the figure. Explain all abbreviations and symbols used in the figure and provide sufficient experimental and statistical detail.
- Provide sufficient experimental detail to allow reproducibility by a person with a similar skill level as yourself.
- Provide details on the origin of chemicals you use. Example: sodium chloride (JT Baker, lot 324)
- If instrumentation is used, report the make and model number of the instruments and location of manufacture. Example: 7500 series High Performance Liquid Chromatography (Agilent; Ames, IA)
- If non-standard software is used, report the version of the software and reference the developer. Example: Sixpack (version 1.57; Webb, 2006).
- Pay attention to significant figures. All measurements should have an uncertainty associated with them. Error only has one significant figure, and this often limits the number of significant figures in a measurement. Example: 3.45 $\pm$ 0.3 should be reported as 3.4$\pm$0.3. If you have questions about this, see your instructor.
- Pay attention to consistent formatting for both in-text citations and in the reference list. Be sure to manually double check that the reference list and text (including figure and tables) have the exact same references.

Review Safety Features in the Lab (REIC 245)

- Cubbies for bags and coats
- Location of personal protective equipment (PPE)
- Safety shower/eyewashes
- Fume hoods
- Exits out of room and out of building
- Waste bottles and broken glass container(s)
- Chemical/safety literature
    - hazard.com/msds is a good start

Laboratory Safety Rules

- USE COMMON SENSE AT ALL TIMES!!!
- No horseplay in lab
- No unauthorized experimentation
- Wear safety glasses/goggles—know when each is required
- Use of correct gloves (when appropriate)
- Knowledge of location and use of MSDS's
- NO food, drink, or gum in lab
- Do not leave fires unattended
- Label all containers with contents, your name, your class, and date/semester
- Report any accident or spill or unsafe condition
- Observe proper storage of chemicals
- Properly disposing of laboratory waste
- Use caution around heat sources, cold sources, flame, electrical equipment
- Properly disposing of glass
- Safely handling chemicals
- No open-toed shoes, no shorts in lab

  Recommend no skirts, hair tied back, no loose sleeves/clothing

Other Important Rules

- This is shared lab space
    - Clean up after yourself
    - Follow storage rules
    - "Default Dirty" Assume benches/glassware is not as clean as you would like.
- Wash your hands frequently
- LABEL EVERYTHING!!!!!
- Use caution if you must transport samples to other rooms
- Inform your instructor and/or TA if you use the last of something or break something
- You are responsible for your own safety, and the safety of everyone else in this room

Good safety = good lab technique = good results

Tentative Schedule (version 1-8-2022)

Date		Monday Lecture	Monday Lab	Wednesday Lab
1-10	1	**Introduction to Measurements (Ch 1)**		
1-17	2			**Noise and Error (Ch 5)** & Student presentation
1-24	3	**Interactions b/w light and matter (Ch 6)**	Standard Addition	Standard Addition
1-31	4	**Components of instruments (Ch 7)** **Atomic spectroscopy (Ch 8-11)**	Standard Addition	Flame AA
2-7	5	**Molecular spectroscopy (Ch 13-15)**	Flame AA	FTIR
2-14	6	**Vibrational Spectroscopy (Ch 16-18)**	FTIR	FTIR
2-21	7	**Mass spectrometry (Ch 20)**	ICP-MS	ICP-MS
2-28	8	**Midterm Exam**	ICP-MS	HPLC
3-7	9	Spring Break		
3-14	10	**Chromatography & HPLC (Ch 26, 28)**	HPLC	HPLC
3-21	11	**GC-MS (Ch 27)**	GC/GC-MS	GC/GC-MS
3-28	12	**NMR (Ch 19)**	GC/GC-MS	GC/GC-MS
4-4	13	**Electron Microscopy (Ch 21-ish)**	NMR	NMR
4-11	14	**X-ray techniques (Ch 12)**	NMR	Tour to EPA NCORE site
4-18	15	**Review of Instruments**	Final presentation	Exam Review
4-25	16			**April 29 (Friday), 1-3 p.m., Final Exam**