GEO 392F/343Q: Fundamentals and Applications of ICP-MS

Fall 2022

Unique Numbers 28079/27819

UT Austin

F 1:00-4:00, Lecture – JGB 3.222 (or on-line); Lab – JGB 1.132

Instructor: *Dr. Nathan Miller,* JGB 6.104a, 471-4810, nrmiller@jsg.utexas.edu
Office hours: W 1-2:30 pm or by appointment, (512) 471-4810

Course Overview: The value of compositional data (isotopic and elemental) to scientific inquiry is indisputable. Such data form the relationships from which trends and processes emerge, enabling prediction and testing of hypotheses. Capable scientists need analytical education in terms of instrumental methods, data generation and evaluation, as well as knowledge of scales of measurement and natural variability. Inductively coupled plasma mass spectrometry (ICP-MS) is widely regarded as the premier technique for trace, minor and major element measurement, and has wide applications in almost every analytical field. This course covers fundamentals of the technique and explores applications and capabilities of ICP-MS through lecture and hands-on labs. Lecture/lab contents are approximately 50:50 with the lab component emphasized in the second half of the course. It is the hands-on experience component of this course, in particular, that facilitates understanding of ICP-MS capabilities.

Tell me and I forget,
Teach me and I may remember *- Xun Kuang (Confucian philosopher)*
Involve me and I'll understand

Prerequisites: Graduate standing in geological sciences or graduate or upper division undergraduate standing and consent of instructor. Working knowledge of MS Excel, including manipulation of data, application of basic algebraic functions to derive statistics, sorting and filtering of data.

Learning Goals: Through understanding of fundamental concepts and processes underlying operation of a modern ICP-MS, successful students completing this course should be able to:

- Engage in practical chemical problem-solving strategies to develop effective analytical methods, particularly where concentration, matrix, interference, and other challenges exist.
- Formulate and make calibration and quality control standards, optimize an ICP-MS, analyze a sample sequence, process raw data to derive concentrations, evaluate data accuracy and precision.
- Critically evaluate ICP-MS method and quality control descriptions in scientific literature.
- Defend the reliability of data resulting from an ICP-MS method of your own design.
- Explain how an ICP-MS works and its pros and cons relative to other analytical techniques.

Evaluation: Pre-lecture readings/Canvas assignments/Class participation – 20%
Mid-term – 20%
Lab exercises and write-ups – 30%
Student analytical method project – 30%

Grading Policy: Your attendance, participation and preparation for class are expected. Assignments are due by the times and dates indicated in the Canvas course syllabus. For schedule conflicts,

contact me well in advance to see if alternative arrangements can be made. Grade boundaries will be determined at the discretion of the instructor to ensure consistency with prior years; the A/B boundary in prior years has typically been in the upper 80's.

Textbook: There are some very good texts on ICP-MS, but I find these to be written at levels well beyond what is required for a basic understanding of ICP-MS. We will instead examine relevant foundational papers documenting aspects of ICP-MS. Course readings will be handed out in class and posted on the course Canvas site.

Canvas: We will use Canvas (https://canvas.utexas.edu/) to post course materials (including reading assignments) and for online discussions. Check your Canvas account and email regularly for class updates. You are responsible for ensuring that the primary email address you have recorded with the university is the one you will check for course communications because that is the email address that Canvas uses. Email is recognized as an official mode of university correspondence; therefore, you are responsible for reading your email for university and course-related information and announcements.

Useful Websites:

- **Plasmachem Listserv** – This listserv is open to the global community of plasma chemists and provides a wealth of practical information on analytical challenges and strategies for coping with complex matrices, interferences, and concentration challenges. You are encouraged to join this for the semester. You will receive daily e-mails that can be quickly surveyed to get a cross-section of analytical challenges and the diversity of ICP-MS applications. To join: **https**://listserv.syr.edu/scripts/wa.exe?SUBED1=PLASMACHM-L&A=1

- **Lab Website:** The ICP-MS lab will be useful: https://www.jsg.utexas.edu/icp-ms/

POLICIES AND RESOURCES

Academic Dishonesty: Academic dishonesty and plagiarism will not be tolerated. You are expected to do your own work in accordance with the UT Honor Code: https://catalog.utexas.edu/law/academic-policies-and-procedures/honor-system/

Sharing of Course Materials is Prohibited: No materials used in this class, including, but not limited to, lecture hand-outs, video recordings, assessments (quizzes, exams, papers, projects, homework assignments), in-class materials, review sheets, and additional problem sets, may be shared online or with anyone outside of the class unless you have my explicit, written permission. Unauthorized sharing of materials is a violation of the University's Student Honor Code and an act of academic dishonesty. Any materials found online that are associated with you, or any suspected unauthorized sharing of materials, will be reported to Student Conduct and Academic Integrity in the Office of the Dean of Students. These reports can result in sanctions, including failure in the course.

Special Needs: The University of Texas at Austin provides upon request appropriate

academic accommodations for qualified students with disabilities. To determine if you qualify, please contact the Division of Diversity and Community Engagement, Services for Students with Disabilities, 471-6259 (https://diversity.utexas.edu/disability/). After your needs are certified, the instructors will work with you to make appropriate arrangements. Special needs requests must be submitted in writing at least a week prior to the affected event, e.g., a test or assignment.

Diversity, Equity & Inclusion: It is our intent that students from all diverse backgrounds and perspectives be well served by this course, that students' learning needs be addressed, and that the diversity that students bring to this class can be comfortably expressed and be viewed as a resource, strength and benefit to all students. Please come to me at any time with any concerns.

Statement on Flexibility, Classroom Safety and COVID-19: As COVID-19 continues to impact the University of Texas at Austin community, we ask that you be respectful of the health and safety concerns and individual circumstances of those around you. With that being said, this semester we commit to being adaptable in this time of great need.

- For any illness, students should stay home if they are sick or contagious, not only to stop the spread, but also to promote their personal wellness.
- The university will continue to provide rapid antigen self-test kits at distribution sites throughout campus. Students can receive up to four tests at a time.
- The university will provide symptomatic COVID-19 testing on campus for all students, faculty, and staff.
- UHS maintains up-to-date resources on COVID, which can be found here: https://www.healthyhorns.utexas.edu/coronavirus.html
    - COVID-19 Information and Resources
    - COVID-19 Exposure Action Chart

If you miss class for an extended period of time due to a personal or family emergency, please see information on Student Emergency Services below.

Overview of Course Assignments

Pre-lecture readings/Class discussion forum assignments (20%): To facilitate engagement outside of lecture and improve your understanding of important concepts brief discussion forum question sets (typically 2/week) based on class readings will be posted on Canvas. Responses are due by the times and dates indicated on the syllabus, and late postings are not allowed. To obtain a full score on each question set, you are asked to:

(1) **Post your thoughtful response to the posed question(s) in Canvas.** Responses should typically be one or more well-crafted paragraphs; aim for a ~300-word response at minimum. Make sure you fully address the question or challenge. Draft these in MS word (or similar) to take advantage of grammar/spelling tools, then paste into Canvas. Do not cut and paste long sections from the assigned readings as quotes. Pose any "murky" questions for which you seek a better understanding.

(2) **Critique a fellow classmate's posting.** After posting, you will be able to see the posts of others. Read and comment substantively to at least one other class posting. Thoughtful critiques should be on the order of a paragraph. You may go back and forth with classmates as much as you want; often these exchanges take on lives of their own and your efforts do show. In your critique, try to build a useful or interesting discussion, for example by:
   - providing further clarification if you feel something important is missing or misunderstood.
   - discussing how the posting influenced your own understanding (for better or worse) or triggered a relevant recollection from lecture or elsewhere.
   - commenting on a murky question or raising new relevant questions.

This grade component will be based on your top 10 scores:

- **5 pts** if (1) and (2) above are complete, thoughtful, substantive, and well-written
- **2.5 pts** if only (1) or (2), but thoughtful and well written
- **0 pts** if no response

Mid-term Exam (20%): Comprehensive and based upon the lecture component, emphasizing ICP-MS fundamentals. Format: multiple choice, short answer, and essay. Class discussion forum topics may be used to pose exam questions.

Lab exercises (30%): Lab exercises are intended to be experiential, allowing time for observation and hands-on participation in ICP-MS analyses. There will be some out-of-class time for evaluations of data sets generated in the lab. Because of the small size of the lab, large classes may be broken down into two separate labs. Many lab exercises will involve friendly team challenges and team learning. Lab exercises are due the following week (see schedule).

- **Max 6 pts/lab** - A total of five (5) labs will be graded.

Class Analytical Project (30%): To develop ICP-MS problem solving skills, you are challenged to develop an analytical method project designed to analyze "real" samples. The project should focus on developing, testing, and documenting quality control for either solution mode ICP-MS or LA-ICP-MS based on analysis of representative samples. Things don't always go as planned and when you run into problems, it is important to learn from them and document the confounding issues (we

don’t want to reinvent the wheel, but it happens with new users). You will document the class project in the form of a website, that you develop over the semester. The project will require analytical time outside of class hours. The sample sets need only be large enough to demonstrate that the methods can obtain high quality data, but we will try to generate interesting datasets of sufficient size that they can be applied to a real research problem.

Grading

- **Proposal - Max 10 pts (draft 5 pts; final 5 pts)**- The class is required to submit a written proposal draft for the method by Tuesday Sep 29, for which I will provide feedback. All required proposal elements must be included in the draft proposal. Final revised proposals incorporating my feedback are due Thursday Oct 29.
- **Sample set preparation** – an extra 5 bonus points if the class is completely ready for both solution mode ICP-MS and LA-ICP-MS by the 12 Nov deadline!!!
- **Class Presentation - Max 10 pts** – We will review the class project website on Zoom during the final exam time slot for the course (likely Th Dec 10, or T Dec 15). You will present your portions of the class method project, as documented on the website. We will rotate through each student, with each sharing their screen of the website and navigating/explaining the content.
- **Final Method Report - Max 10 pts** - You will submit a written summary of your analytical method and quality control in the form of a methods section/appendices from a peer-reviewed journal. The written summary should logically follow from the project proposal and is due on the day of Final Presentations (TBD, likely Th Dec 9, or T Dec 14).

Class Schedule of Activities

Wk	Tue	Thu	Fri			Lecture/Lab Topic	Discussion Forums	Labs	Method Project
1			26-Aug	FUNDAMENTALS	1	ICP-MS vs. the world!			
					2	Heritage of ICP-MS			
2	30-Aug						Topic 1 due		
		1-Sep					Topic 2 due		
			2-Sep		3	Atomic structure, isotopes, ions, ionization; Overview of ICP-MS components			
					4	Nebulizer-Spray Chamber, Torch			
3	6-Sep						Topic 3 due		
		8-Sep					Topic 4 due		
			9-Sep		5	Plasma Source			
					6	Vacuum Interface			
4	13-Sep						Topic 5 due		
		15-Sep					Topic 6 due		
			16-Sep		7	Ion Focusing System			
					8	Quadrupole Mass Analyzer			
5	20-Sep						Topic 7 due		
		22-Sep					Topic 8 due		
			23-Sep		9	Other Mass Analyzers, Magnetic Sectors, Time-of-Flight			Proposal draft due
					10	Spectral Interferences: Isobaric, Polyatomic, Doubly-Charged			
6	27-Sep						Topic 9 due		
		29-Sep					Topic 10 due		
			30-Sep		11	Collision-Reaction Cell (CRT) Technology			
					12	Detectors			
7	4-Oct						Topic 11 due		
		6-Oct					Topic 12 due		
			7-Oct		13	Sample Prep & Contamination Control			
					14	Analysis of solid materials by LA-ICP-MS			
8	11-Oct						Topic 13 due		
		13-Oct					Topic 14 due		
			14-Oct		15	L.A.S.E.R., laser system, laser projects			
					16	Murky Questions and Midterm Review			
9	18-Oct					Study for midterm			
		20-Oct				Study for midterm			
			21-Oct		17	**MIDTERM EXAM (20%)**			
				APPLICATIONS	18	Lab Tour, Tuning, Lab 1 - Unknown identification - Full Spectral Mass Scans			
10	25-Oct								
		27-Oct						Lab 1 due	
			28-Oct		19	Lab 2 - Unknown identification - Semi-quant analysis			Final Proposal due
					20	Water collection FT; quantitative solution mode analysis			
11	1-Nov								
		3-Nov						Lab 2 due	
			4-Nov		21	Making calibration standards			
					22	Lab 3 - Calibration standard making			
12	8-Nov								
		10-Nov						Lab 3 due	
			11-Nov		23	Lab 4 - Solution mode data reduction			Sample Prep Complete
					24	LA-ICP-MS demo/Iolite data reduction/Lab 5 - Longerich et al. - data reduction			
13	15-Nov								
		17-Nov				Scheduled method project development time		Lab 4 due	
			18-Nov						
14	22-Nov								
		24-Nov				*Fall/Thanksgiving Break (no class)*			
			25-Nov						
15	29-Nov								
		1-Dec				Scheduled method project development time		Lab 5 due	
			2-Dec						
16	6-Dec								
		8-Dec							
			9-Dec			**METHOD PROJECT PRESENTATIONS**			Website Review

GEO 392F/343Q - Class Method Proposal

Purpose

The purpose of your methods project is to develop, refine, and defend an ICP-MS analytical method capable of obtaining high quality elemental or isotopic compositions, based on your understanding of ICP-MS capabilities. Methods may be either solution mode or laser ablation mode, but must be applied to an analytical challenge on real world samples. ***Proof of the method capability is by demonstrating that high quality data were obtained on a representative sample set.***

Your method proposal should document the plan you expect to follow to develop and test your ICP-MS or LA-ICP-MS method. It should also demonstrate an understanding of the implications of previous relevant work, such as any anticipated challenges for the analytes of interest (isotopes you wish to measure). Your proposal should convince the reviewer (me) that your method development plan is realistic and has the potential to obtain useful results. Proposals should be concise, compelling and well worded to establish credibility.

Graded Proposal Elements (10 pts/ea):

1. Title Page – should
    - Concisely identifies the specific subject in as few words as possible
    - Attracts attention to the research hypothesis
    - Clearly reflects the method development objectives
2. Executive summary/Abstract
    - Describes the proposed research objective or hypothesis
    - Describes the method objectives & expected outcomes
3. Research objective or hypothesis and specific objectives of the method
    - Briefly elaborates the greater research objective that your method will be applied to in order to provide useful data. Elaborates in more detail what the method will do and specifies all analytes to be included.
4. Discussion of significance or need (justification)
    - Describes why the method is of potential importance and why analytes listed above are specifically important or relevant to the research objective(s).
    - Describes what degree of accuracy and precision are required for the method to be useful.
    - May present previous or preliminary research bearing on the need for method development.
5. Review of relevant work (literature review)
    - Summarizes relevant ICP-MS or LA-ICP-MS research applied to similar sample types; includes recent and state-of-the-art research applicable to your research goal; PlasmaChem Listserv is a possible resource for finding published research
    - Describes any likely analytical challenges for the analytes in your method (e.g., interferences, matrix effects, calibration) and how they have been addressed in previous work
6. Materials and methods
    - Outlines the working plan for how you will develop and test your method in order to obtain the required data quality. States what you plan to analyze and how you will evaluate accuracy and precision of the data obtained.
    - This section should justify the budget materials and time described separately in Section 8 below.
    - REMEMBER – the point of this project is to develop a method and prove it works well, so you need only run a reasonable number of samples. Do not propose to run 100s of samples.
7. Discussion of possible outcomes
    - Enumerates potential benefits to be derived from the final method; draws the reader back to the research question, hypothesis, and objectives
8. Timeframe and budget
    - Time and money are often vital; you must convince grantors (ME) that your plan is realistic for the proposed research. Some method development and analysis time outside of class will be necessary.
    - Although you will not be charged for your class method project, provide a budget based on $17 per solution mode ICP-MS analysis (inclusive of all calibration standards, quality control standards, blanks, spikes, and unknowns) and $73/hr for LA-ICP-MS time.
9. Biography of investigators
    - This is an abbreviated resume that indicates that you are capable of developing the proposed method; emphasizes relation of applicable training to expertise needed; avoids points unimportant to this research.
10. References
    - This section indicates the extent to which you've explored relevant analytical and non-analytical literature for your subject – you need both. Try to find the best and most recent analytical references (solution mode ICP-MS or LA-ICP-MS) references relevant to analysis of similar samples or analytes. Also include relevant non-analytical background references.