GEO 391/343Q: Fundamentals and Applications of ICP-MS

TTh 2:00-3:30, Lecture – JGB 3.116; Lab – JGB 1.132

Fall 2018 UT Austin

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

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 advancement of hypotheses. Capable scientists need analytical education in terms of instrumental methods, data generation and evaluation, and 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, through hands-on labs, applications and capabilities of ICP-MS. 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.

I hear and I forget I see and I hear, I remember I see, I hear **and I do**, I understand - Old Chinese proverb

Prerequisites: Graduate standing in geological sciences or graduate standing and consent of instructor. Working knowledge of MS Excel, including manipulation of rows and columns 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 introduced through lecture, and hands-on experience/skills developed in lab, successful students completing this course should be able to:

- Articulate how an ICP-MS works and explain its pros and cons relative to other analytical techniques.
- Engage in practical chemical problem-solving strategies with respect to the capabilities of ICP-MS, in order to develop an effective analytical method, 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 and evaluate 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 in a presentation, thesis/masters defense, or peer discussion.

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

Grading Policy: Your attendance, participation and preparation for class are expected. Assignments are due by class meeting time on the 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.

Required and recommended materials: 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. Rather than refer to a single text, we will instead examine relevant foundational papers documenting aspects of ICP-MS. Course readings will be made available in pdf form on the course Canvas site.

PLASMACHEM LISTSERV – This is a very useful listserv open to the global community of plasma chemists. The plasmachem archives provide a wealth of practical information about 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 numerous e-mails daily and these can be quickly surveyed to get a cross-section of analytical challenges and the diversity of ICP-MS applications. To join: https://answers.syr.edu/pages/viewpage.action?pageId=918117

Class Websites: We will use Canvas throughout the semester: <u>https://courses.utexas.edu/</u> I will try to adhere to the course schedule as much as possible, but some modifications are likely for logistical reasons. Any such changes will be communicated weekly and updated in the Canvas syllabus. The research web site for the JSG ICP-MS lab will also be useful: <u>http://www.jsg.utexas.edu/icp-ms/</u>

Notice: Students with disabilities may request appropriate academic accommodations from the Division of Diversity and Community Engagement, Services for Students with Disabilities, 471-6259, http://diversity.utexas.edu

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: <u>http://www.engr.utexas.edu/undergraduate/forms/462-university-of-texas-honor-code</u>

Overview of Course Requirements/Assignments

Pre-lecture readings/Class discussion forum assignments (20%): Let's face it – it's a long time between Thursday and the following Tuesday. To facilitate engagement outside of lecture, weekly readings and other materials will be made available in class and/or through the Canvas course site. Brief question sets (typically 2/week; 12-14/semester) based on readings will be posted each week on Canvas, which are due before the next week (by class time on Monday). Your top ten scores will be used to calculate your final grade for this component of the course.

For each question set, you are asked to do two things to obtain a full score:

(1) Publish your thoughtful responses to these as part of a class forum. The forum is designed to improve your understanding of important concepts, and also to pose questions to the class for concepts you seek a better understanding of.

(2) Critique at least one other posting by a fellow classmate and/or pose a "murkiest question" to the class. In your critique, you might provide further clarification (if you feel something important is missing or misunderstood) or discuss how their posting influenced your greater understanding (for better or worse). You may go back and forth as much as you want; often these exchanges take on lives of their own.

• **Grading: Max 5 pts/question set** – if complete, thoughtful, and well written; all questions addressed (4 pts) and murky question posed and/or answered (1pt).

Mid-term Exam (20%; Tue 23-Oct): Comprehensive and based upon the lecture component, emphasizing

ICP-MS fundamentals. Format: multiple choice, short answer, and essay. The class forum may be used to pose questions based on "murkier" concepts or extended forum topics.

Student Analytical Project (30%): To develop ICP-MS problem solving skills, you will be challenged to develop a solo analytical method toward a limited project in your area of interest/specialization. The method should be outside your comfort zone (if you are already a regular user of ICP-MS data) and should include an analytical challenge (e.g. elements with masses between 40 and 80). Your solo project will require analytical time outside of class hours, and involve analysis of "real" samples. Sample sets need only demonstrate that the method works and obtains high quality control; e.g. 5-15 samples are adequate to demonstrate quality control. You are encouraged to develop an interesting data set, but do not attempt to analyze huge sample sets. *Important deadlines:*

- Tuesday Oct 2 Rough draft of project proposal due
- Thursday Nov 1 Final draft of project proposal due
- Tuesday Nov 20 Sample preparation complete; samples must be fully ready for analysis.

Projects are about the method, and its development, to optimize analyses of the samples of interest.

- **Proposal Max 10 pts (draft 5 pts; final 5 pts)** You are required to submit a written proposal for the solo method by Tuesday Oct 2, for which I will provide feedback toward development of a revised final proposal. Required proposal elements will be posted on Canvas. Final revised proposals are due Thursday Nov 1.
- Class Presentation Max 10 pts You will present your solo method concept, development, and results to the class during the final week of the course in the form of a short GSA type presentation (10-12 minutes followed by a few minutes for questions).
- 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 by Tuesday Dec 18.

Lab exercises (30%): Lab exercises are intended to be experiential, and to allow time for observation and hands-on participation in ICP-MS analysis. There will be some out-of-class time for evaluations of lab data sets, which will be posted on Canvas. Because of the small size of the lab, large classes may be broken down into two separate labs. There will be some team lab exercises involving a lab partner. Lab exercises may be modified but write-ups are due the following week.

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

Class Schedule of Activities

Wk	Day	Date	Торіс		Lecture/Lab	Meeting Location	Important Dates
1	Th	30 - Aug		Intro & Chem Bkgd; IC MS components	1. Course Introduction Pt 1: ICP-MS vs. the world	JGB 3.116	
2	т	4-Sep			1. Course Introduction Pt 2: Heritage of ICP-MS		Discussion topic 1 due
	Th	6-Sep			2. Atomic structure, isotopes, ions, ionization; Overview of major ICP-MS instrument components		Discussion topic 2 due
3	т	11-Sep	Fundam	Ionization	3. Sample Introduction: Nebulizer-Spray Chamber-Torch		
	Th	13-Sep			4. Sample Introduction: Plasma Source		Discussion topic 3 due
4	т	18-Sep		Ion Sampling	5. Sample Introduction: Vacuum Interface 6 Focusing System		Discussion topic 4 due
	Th	20-Sen		Ion Filtering & Interference Removal Strategies	7. Quadrupole Mass Analyzer		Discussion topic 5 due
5	т	25-Sen			8. Other Mass Analyzers: Magnetic Sectors, Time-of-Flight		Discussion topic 6 due
	Th	27-Sen			9. Spectral interferences: isobaric, polyatomic and doubly charged species: Mathematic correction equations		Discussion topics 7 due
6	т	2-Oct			10. Collision/Reaction Cell technology		Project Proposal Draft 1 Due
	Th	4-Oct		Ion Detection	11. Detectors		Discussion topic 8 due
7	т	9-Oct		Sample Prep, QA/QC	12. Sample Prep Considerations & Contamination Control		Discussion topic 9 due
,	Th	11-Oct		Laser Ablation	13. Analysis of solid materials by LA-ICP-MS		
8	Т	16-Oct			14. Analysis of solid materials by LA-ICP-MS		Discussion topics 10-11 due
	Th	18-Oct		Midterm Review	15. Murky questions and review of topics		Discussion topic 12 due
9	т	23-Oct			Midterm Exam		Midterm Exam
	Th	25-Oct	lic	Solution mođening, unknown ID via FSMS & semiquant analysis, making cal stds, methoo design for quant analysi & data reduction	Lab 1 Lab tour; Tuning the Instrument; Solution mode: tear unknown identification via full spectral mass scans	JGB 1.132	
10	т	30-Oct			Lab 2.Solution mode: unknown identification via semi-qua analysis		Discussion topics 13-14 dutAB 1 Due
	Th	1-Nov			Water collection FT; Quantitative solution mode analysis		Final Proposal Due
11	т	6-Nov			Lab 3.Solution mode: making calibration standards		LAB 2 Due
	Th	8-Nov			Team calibration standard making, testing		
12	т	13-Nov			Lab 4.Solution mode data reduction		LAB 3Due
	Th	15-Nov		LA-ICP-MSming, exp design, analysis, data reduction	LA-ICP-MS demo and Iolite		
13	т	20-Nov			Lab 5.LA-ICP-MS data reduction	JGB 3.116	LAB 4 Due; Project Sample Preparation Complete
	Th	22-Nov	Ann	Hands-On Student Project Time	Thanksgiving Break November 21-24 (no class)	JGB 1.132	
14	т	27-Nov			Scheduled student individual project time		LAB 5 Due
	Th	29-Nov			Scheduled student individual project time		
15	Т	4-Dec			Scheduled student individual project time		
	Th	6-Dec			Scheduled student individual project time		
16	т	11-Dec.		Student project presentations	Student presentation and final paper preparation (no class JGB 3		Finals Dec 11, 13, 18 Student project presentations Final project paper due
als	Th	13-Dec				JGB 3.11	
Fin	т	18-Dec			Student presentations and final project paper due		