GEO 391: Fundamentals and Applications of ICP-MS

Fall 2017

MW 1:30-3:00, Lecture – JGB 3.120; Lab – JGB 1.132 UT Austin

Instructor: *Dr. Nathan Miller,* Office**:** JGB 6.104a, Office Hours: T 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: https://courses.utexas.edu/
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: http://www.jsg.utexas.edu/icp-ms/

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

Overview of Course Requirements/Assignments

Pre-lecture readings/Class discussion forum assignments (20%): Let's face it – it's a long time between Wednesday and the following Monday. 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%; Mon 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:***

- **Mon Oct 2 – Rough draft of project proposal due**
- **Wed Nov 1 – Final draft of project proposal due**
- **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 Mon 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 Wed 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 Mon 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		Topic	Lecture/Lab	Meeting Location	Important Dates	Classes	
1	W	30-Aug	Fundam	Intro & Chem Bkgd; ICP-MS components	1. Course Introduction, ICP-MS vs the world			1	
2	M	4-Sep			**Labor Day (no class)**				
	W	6-Sep			2. Atomic structure, isotopes, ions, ionization; Overview of major ICP-MS instrument components		Discussion topic 0&1 due	2	
3	M	11-Sep		Ionization	3. Sample Introduction: Nebulizer-Spray Chamber-Torch			3	
	W	13-Sep			4. Sample Introduction: Plasma Source		Discussion topics 2&3 due	4	
4	M	18-Sep		Ion Sampling	5. Sample Introduction: Vacuum Interface 6. Focusing System			5	
	W	20-Sep		Ion Filtering & Interference Removal Strategies	7. Quadrupole Mass Analyzer		Discussion topics 4&5 due	6	
5	M	25-Sep			8. Other Mass Analyzers: Magnetic Sectors, Time-of-Flight	JGB 3.120		7	
	W	27-Sep			9. Spectral interferences: isobaric, polyatomic and doubly charged species; Mathematic correction equations		Discussion topics 6&7 due	8	
6	M	2-Oct			10. Collision/Reaction Cell technology		**Project Proposal Draft 1 Due**	9	
	W	4-Oct		Ion Detection	11. Detectors		Discussion topics 8&9 due	10	
7	M	9-Oct		Sample Prep, QA/QC	12. Sample Prep Considerations & Contamination Control			11	
	W	11-Oct		Laser Ablation	13. Analysis of solid materials by LA-ICP-MS		Discussion topics 10&11 due	12	
8	M	16-Oct			14. Analysis of solid materials by LA-ICP-MS			13	
	W	18-Oct		Midterm Review	15. Murky questions and review of topics		Discussion topics 12&13 due	14	
9	M	23-Oct		**Midterm Exam**			**Midterm Exam**	15	GSA 10/22-25
	W	25-Oct	Applic	Solution Mode Tuning, Interference Removal, Data Reduction	Lab tour, Tuning Instrument (No Gas), Measuring isotope abundances in different matrices I	JGB 1.132		16	
10	M	30-Oct			CRC tuning (He & H2); evaluating isotope abundances II; Collection & analysis of waters		**LAB 1 Due**	17	
	W	1-Nov			Solution mode data reduction; Tindelpina Shale practice data set		**Final Proposal Due**	18	
11	M	6-Nov		Laser Mode	LA-ICP-MS demo and Iolite; Lab 2 water analyses data reduction (graded)		**LAB 2 Due**	19	
	W	8-Nov		Calibration	Solution mode: making calibration standards			20	
12	M	13-Nov		Unknown identification	Solution mode: unknown identification via full spectral mass scans		**LAB 3 Due**	21	
	W	15-Nov			Solution mode: unknown identification via semi-quant analysis			22	
13	M	20-Nov		LA-ICP-MS data reduction	LA-ICP-MS data reduction	JGB 3.120	**LAB 4 Due; Project Sample Preparation Complete**	23	
	W	22-Nov		Hands-On Student Project Time	**Thanksgiving Break November 22-25 (no class)**	JGB 1.132			
14	M	27-Nov			Scheduled student individual project time		**LAB 5 Due**	24	
	W	29-Nov			Scheduled student individual project time			25	
15	M	4-Dec			Scheduled student individual project time			26	
	W	6-Dec			Scheduled student individual project time			27	
16	M	11-Dec		Student project presentations	Last day of classes	JGB 3.120	**Finals Dec 8-10, 12-14**	28	AGU 12/11-15
Finals	W	13-Dec			**Student project presentations**		**Student project presentations**		
	M	18-Dec			**Final project paper due**		**Final project paper due**		