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

Unique Numbers 29295/29005

T, Th 12:30-2:00, Lecture: JGB 3.222, Lab: MBB 1.318

Fall 2025 UT Austin

Instructor: Dr. Nathan Miller, MBB 1.318, 512-471-4810, nrmiller@jsg.utexas.edu

Office hours: W 1-2:30 pm or by appointment

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 scientific 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
Involve me and I'll understand

- Xun Kuang (Confucian philosopher)

**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 ICP-MS 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, go to <a href="https://listserv.buffalo.edu/">https://listserv.buffalo.edu/</a>, select "Subscribe or Unsubscribe", and enter "PLASMACHEM-L" for the name of the list to join. You then are asked to provide your name, e-mail, and to register a password. Once you have provided the basic info, you will begin receiving listserv e-mail from the global community of ICP-MS users. Here is a pdf with instructions for subscribing.
- Lab Website: The ICP-MS lab will be useful: https://www.jsg.utexas.edu/icp-ms/
- Interactive Periodic Table: <a href="https://www.inorganicventures.com/periodic-table">https://www.inorganicventures.com/periodic-table</a>
- **Molecular Weight Calculator:** <a href="https://www.lenntech.com/calculators/molecular/molecular-weight-calculator.htm">https://www.lenntech.com/calculators/molecular/molecular-weight-calculator.htm</a>
- GeoREM Geological and Environmental Reference Materials: <a href="http://georem.mpch-mainz.gwdg.de/start.asp?dataversion=current">http://georem.mpch-mainz.gwdg.de/start.asp?dataversion=current</a>

### **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: <a href="https://catalog.utexas.edu/law/academic-policies-and-procedures/honor-system/">https://catalog.utexas.edu/law/academic-policies-and-procedures/honor-system/</a>

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.

Artificial Intelligence: All work in this course must be your own or created in group work, where allowed. The use of generative artificial intelligence tools (or Large Language Models [LLMs]) such as CoPilot or ChatGPT in this class is permitted on a limited basis. Generative AI should be used with caution and proper citation, as the use of generative AI must be properly attributed. Failing to cite generative AI use according to the citation policy in this course, even where permitted, may constitute a violation of UT Austin's Institutional Rules on academic integrity and may be referred to the Office of Student Conduct and Academic Integrity for resolution.

**Citation Policy:** In-text citations must be used when you present information obtained from external sources. Peer-reviewed publications from scientific literature should be cited over websites or Al summaries. Summarize content from original sources in your own words and avoid using direct quotes. We will use the citation standard for the Geologic Society of America, as it is commonly used in geoscience for in-text citations and references cited. GSA reference guidelines and examples (pdf) can be found here (https://guides.library.sc.edu/ld.php?content\_id=50991215).

**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 Student Affairs, Disability and Access, 512-471-6259 (<a href="https://disability.utexas.edu/accommodations-and-services/">https://disability.utexas.edu/accommodations-and-services/</a>). 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.

**Statement on Flexibility and Classroom Safety:** Be respectful of the health and safety concerns and individual circumstances of those around you.

- 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.
- If you must miss class for an extended period of time due to a personal or family emergency, please contact me.

## **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 canvas, 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 to the class 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 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.; late labs docked 1 pt (17%)/day

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. Group projects with 2-3 students are possible. 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 method you've developed 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

• Nov 6 - Sample Preparation Due – 5 pts. This is an easy one. Just have your samples in the lab and completely ready to analyze by Tuesday November 5. Plan accordingly to meet this deadline. For solution mode ICP-MS projects, this means having your liquid samples collected and ready to dilute in the lab for analysis. For LA-ICP-MS projects, this means having your appropriately prepared samples collected and ready to load (with appropriate standards) into the sample cell of the laser system.

Why is this one listed first? Because almost every semester this deadline bites one or more students. Why give away 5% of the grade? If you need to find/acquire samples or have some specialized sample preparation for your methods project, formulate a plan and act well before the deadline. Missed deadlines often involve time delays related to locating samples (e.g., from a busy faculty member or graduate student), projects requiring some sort of specialized digestions (e.g., silicate rocks require HF digestions that cannot be performed in the ICP-MS lab complex), sample preparation (e.g., sending out samples to have thin sections made, separating minerals to make polished epoxy mounts), or projects requiring a precursor analysis (e.g., you have an ongoing project and you want to analyze some component of it after processing/experiments).

- **Sept 25 Proposal Rough Draft Due 5 pts.** This will be in the form of a website. This content will ultimately end up in your final Method Project website. I will provide feedback toward the final draft.
- Oct 23- Final Proposal Due 5 pts. Final revised proposals (in website form) incorporating
  my feedback are due Tuesday Oct 22.
- **Dec 11 Final Method Website Published- 10 pts.** I will provide guidelines regarding essential site content.
- **Dec 11 Class Presentation 5 pts.** You will present to the class your final website documenting your method project and the data quality obtained.

# **Class Schedule of Activities**

Wk	Day	Date		Topic	Lecture/Lab	Meeting Location	Important Due Dates	Classes
1	т	26-Aug		Intro & Chem Bkgd; ICP- MS components	0. Course Introduction; ICP-MS vs. the world	JGB 3.222		1
					1. Course Introduction Pt 2: Heritage of ICP-MS		Discussion topic 1	
	Th	28-Aug			2. Atomic structure, isotopes, ions, ionization; Overview of		Discussion topic 2	2
2	Т	2-Sep		lonization	major ICP-MS instrument components  3. Sample Introduction: Nebulizer-Spray Chamber-Torch		· · · · · · · · · · · · · · · · · · ·	3
	Th	4-Sep			4. Sample Introduction: Plasma Source		Discussion topic 3	4
3	Т	9-Sep	Fundamentals		Sample Introduction: Plasma Source     Sample Introduction: Vacuum Interface			5
	Th	11-Sep		Ion Sampling	6. Ion Focusing System		Discussion topic 4	6
4	Т	16-Sep		Ion Filtering & Interference Removal Strategies	Quadrupole Mass Analyzer     Spectral interferences: isobaric, polyatomic and doubly		Discussion topic 5	7
	Th	18-Sep	ame		charged species; Mathematic correction equations		Discussion topic 6	8
5	Т	23-Sep	Fund		9. Collision/Reaction Cell Technology		Discussion topics 7	9
	Th	25-Sep			10. Spectral interferences and Tandem ICP-MS/MS (aka Triple Quadrupole ICP-MS) - the latest and greatest		Web Draft of Method Project Proposal	10
6	Т	30-Sep		Ion Detection	11. Detectors		Discussion topic 8	11
	Th	2-Oct		Sample Prep, QA/QC	12. Sample Prep Considerations & Contamination Control		Discussion topic 9	12
7	т				13. Analysis of solid materials by LA-ICP-MS		Discussion topic 10	
		7-Oct		Laser Ablation	Laser Ablation  14. Analysis of solid materials by LA-ICP-MS		Discussion topic 11	13
	Th <b>F</b>	9-Oct <b>10-Oct</b>		Optional Midterm Review	Murky questions and review of topics			14
8	Т	14-Oct			Midterm Exam		Midterm Exam	15
	Th	16-Oct		Solution mode	Lab tour; using pipettes, making gravimetric dilutions		Discussion topic 12	16
9	Т	21-Oct		ICP-MS:	Lab 1. Solution mode data reduction		Discussion topic 13	17
	Th	23-Oct		Tuning, unknown identification via full	Lab 2. LA-ICP-MS data reduction		Final Method Project Proposal Published on Website	18
40				spectral mass scans & semiquant analysis,	Lab 3-4. Solution mode: unknown identification via Full Spectral Mass Scans and Semi-quant Analysis		Discussion topic 14  LAB 1	
10	T	28-Oct	Applications	making cal stds, method design for quant analysis, & data reduction	Lab 5. Solution mode: making calibration standards	- MBB 1.318	LAB 2	19
	Th	30-Oct			Team calibration standard making, testing		LAB 3	20
11	Т	4-Nov			LA-ICP-MS demo and lolite		Project Sample Prep Complete	21
	Th	6-Nov		Tuning, experiment	Ex for the demodratione		LAB 4	22
12	Т	11-Nov		reduction Hands-On				23
	Th	13-Nov		Class Project Time	Scheduled class project time		LAB 5	24
13	Т	18-Nov		Hands-On Class Project Time	Scheduled class project time			25
		20-Nov		Hands-On Class Project Time	Scheduled class project time			26
				Thanksgivin	g Break November 24-29 (no class)			
14	Т	2-Dec		Hands-On Class Project Time	Scheduled class project time			27
	Th	4-Dec		Hands-On Class Project Time	Scheduled class project time	MBB 1.318		
15				Hands-On	Scheduled class project time (optional, if necessary)			28
Finals	Т	9-Dec		Class Project Time	thod project presentations	MBB 1.318	Final Project Websites Posted	29
Œ	Th	11-Dec		14161			s roject ressites rosted	30

## GEO 392F/343Q - Class Method Proposal

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 method. It should also demonstrate an understanding 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. Grey shaded topics below show the four separate web pages for your proposal.

### Graded Proposal Elements (10%/ea):

- 1. Title Page This can be your website home page. It should
  - Title should concisely identify the specific subject in as few words as possible, attract attention to the research hypothesis, and clearly reflect the method development objectives. Page should include a "killer" graphic to attract reader interest. Title should be along the lines of developing a method to determine . . . by LA/ICP-MS.

#### Proposal - should include Items 2-8 below

- 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-ofthe-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. Use GSA guidelines for in-text citations (see syllabus).
- 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 \$20 per solution mode ICP-MS analysis (that's \$20 for every calibration standard, quality control standard, blank, spike, and unknown analyzed) and \$65/hr for LA-ICP-MS time.
- 9. Biography of investigators (separate web page)
  - 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. It should be written in 3<sup>rd</sup> person voice (Jane Doe is a first-year doctoral student in the Department of . . .).
- 10. References (separate web page)
  - This section indicates the extent to which you've explored relevant analytical and non-analytical literature for your subject you need both. Include 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. Use the GSA citation guidelines for formatting (see syllabus).