GEO 390S: Analytical Methods in Mass Spectrometry

Spring 2013
MW 1-2, Lecture – EPS 4.104; F 12-3, Lab – various JGB labs
UT Austin

Instructors:
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Course Overview: Mass spectrometers are essentially analytical balances that operate at molecular and atomic levels. Compositional data (both isotopic and elemental) provided by mass spectrometric techniques are invaluable to many lines of scientific inquiry. Many types of mass spectrometers have been developed to suit a wide range of applications (>10 in the Jackson School of Geosciences!), but all involve the conversion of sample molecules into charged particles (ions) that are then measured according to their mass-to-charge ratio to provide specific information about chemical identity and abundance. This course provides an introduction to inorganic mass spectrometry methods and their applications to the Earth sciences. Five key modalities are surveyed (TIMS, ICP-MS, LA-ICP-MS, MC-ICP-MS, and IRMS) in lecture, with hands-on experience provided in weekly labs. Knowledge and skills developed in this course are intended to provide an understanding of how mass spectrometers work, how these techniques are used in geoscience research, and what is necessary to generate and critically evaluate high-quality data.

"At first there were very few who believed in the existence of these bodies smaller than atoms. I was even told long afterwards by a distinguished physicist who had been present at my [1897] lecture at the Royal Institution that he thought I had been 'pulling their legs." -J.J. Thomson (1936)

Prerequisites: Graduate standing in geological sciences, upper division undergraduate standing in geological sciences and consent of instructor, or graduate standing in another field and consent of instructor.

Learning Goals: ***The overarching goal of this course is for students to become critical consumers and producers of data using inorganic mass spectrometry techniques.*** Through understanding of fundamental concepts and processes underlying operation of modern mass spectrometers introduced through lecture, and hands-on experience/skills developed in lab, successful students completing this course should be able to:

- Explain basic principles of operation for each mass spectroscopic technique
- Understand basic sample preparation considerations for each technique
- Articulate how instrumental parameters are optimized in order to obtain robust operating conditions
- Describe the strengths and limitations of each mass spectroscopic technique
- Understand how raw data generated by each technique should be processed to obtain high quality data (corrections for drift, mass fractionation, dilution)
- Calculate and evaluate important figures of merit to evaluate analytical quality (accuracy, precision, mass resolution, abundance sensitivity)
- Understand how the various techniques complement each other and can be used to evaluate geoscience problems (geologic applications of the techniques)

Evaluation:	Class participation/engagement	5%
	Mini-quizzes (top 10 scores)	10%
	Discussion forum	15%
	Lab exercises and write-ups	40%
	Final synthesis 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 Blackboard course syllabus. For schedule conflicts, contact us well in advance to see if alternative arrangements can be made.

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

Class Websites: We will use Blackboard throughout the semester: https://courses.utexas.edu/
We will try to adhere to the course schedule as much as possible, but some modifications are possible for logistical reasons. Any such changes will be communicated weekly and/or updated in the Blackboard syllabus.

SERC Geochemical Instrumentation and Analysis website provides information on mass spectrometry as well as other geochemical techniques.
http://serc.carleton.edu/research_education/geochemsheets/index.html

The research website for the JSG ICP-MS lab will also be useful:
https://webspace.utexas.edu/wg3486/geo-web-folders/miller/QuadICPMSlab/ICP-MS/Home.html

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://www.utexas.edu/diversity/ddce/ssd/

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://registrar.utexas.edu/catalogs/gi09-10/ch01/index.html

Overview of Course Requirements/Assignments

Class participation/engagement: It is important to be prepared for each lecture and lab. You should critically read assigned reading materials and be prepared for class discussions. Because there are only a few (4-6) lectures and (1-2) labs per mass spectrometry technique, it is vital to regularly attend class. The Blackboard activities, described below, are designed to help keep you engaged outside of class.

Weekly mini-quizzes (Wednesdays): Let's face it, lectures are short and there is a lot of time to forget between classes. To promote engagement and help reinforce lecture content, there will be a weekly "mini quizzes" based on reading assignments. The quizzes, worth 10 pts each, are hosted on Blackboard, and must be taken at least 1 hour prior to class. There will be >10 mini quizzes during the semester, of which your top 10 scores will count toward this component of your grade.

Weekly discussion forum (Mondays): As we survey each MS technique, you are required to populate a discussion forum (Blackboard) by responding to weekly threads that are synced to lecture and/or lab material. The threads provide an opportunity for you to articulate your understanding of what is important for all aspects of a mass spectrometric analysis, in addition to critiquing the content of other student responses. Your first critique should be on a thread response that has not previously been responded to, however you may respond to as many thread responses as you wish. Thread responses must be submitted at least 1 hour prior to class (Blackboard time stamp) on the assigned date for full credit or they are considered late and worth half-credit. Grading criteria are:

- 3 pts — Full thoughtful response to thread and at least one other classmate; responses are clear, concise and well written.
- 2 pts — Response to original thread and at least one other classmate made, but responses are incomplete, vague, or poorly written.
- 1 pt — Response to only original thread or only one classmate, regardless of content and quality
- 0 pts — no submission (please don't do this!!!)

Lab exercises: Lab exercises are intended to be experiential, and to allow time for observation and hands-on participation. There will be out-of-class time required for evaluations of lab data sets, and participation may also be through Blackboard. ***All labs, unless otherwise stated, are due (submitted) by the beginning of class on the Wednesday after they are assigned.**

Final synthesis project: You will demonstrate that you have become a critical consumer of mass spectrometric data by summarizing your knowledge in three ways.

1) Methods sections (75 pts) - During each module, you will be given a data set generated using the mass spectrometer presented during that module. You will arrange the data as if you were preparing a publication. Create a data table using the data and write a methods section explaining how the data was collected. It is important to include all the necessary measures of data quality. We strongly recommend that you complete each data table and methods section as you complete each module (15%/module)
   Friday March 8 Road Check – TIMS and ICP-MS methods drafts due
2) Comparison (15 pts) - You will prepare a short essay comparing and contrasting the 5 modalities presented in this class, based on a set of questions that will be provided to you during the first week of class. These questions will be addressed during each module and will be part of the forum discussion on Blackboard.
3) Personal impact (10 pts) - Describe how what you have learned in this class impacts your research. This question is intentionally open-ended as we want you to reflect on how you might apply what you have learned in this class.

Schedule of Class Activities

Wk	Date	Day	Method Topic (Instructor)	Class/ Graded Labs	Lab Location	Class Topic
1	14-Jan	M	Intro (Miller/Loewy)	Lecture		Course outline, grading, deliverable, basics of mass spectrometry
1	16-Jan	W		Lecture		What are mass spectrometers
1	18-Jan	F		Lecture		Metrology - science of measurement vs. mass spectrometry
2	21-Jan	M	***MLK Jr Day - Holiday***			
2	23-Jan	W		Lecture		Ions, Tigers and Bears . . . Oh my!!! Elements, atoms, isotopes, ions
2	25-Jan	F		Lecture		Sample collection, preparation, quality control, data integrity
3	28-Jan	M	TIMS (Loewy)	Lecture		What is TIMS? How does it work?
3	30-Jan	W		Lecture		Applications of TIMS, strengths and weaknesses
3	1-Feb	F		Lab Tour	JGB 1.126	The Clean Lab - column chromatography - chemical separation
4	4-Feb	M		Lecture		Measuring ratios (mass fractionation)
4	6-Feb	W		Lecture		Determining concentrations (isotope dilution)
4	8-Feb	F		**Lab 1**	JGB 1.126	The Triton - Running the machine
5	11-Feb	M		Lecture		How do we used standards?
5	13-Feb	W		Lecture		Uncertainties and sources of error
5	15-Feb	F		**Lab 2**	JGB 1.126	The Triton - Collecting data
6	18-Feb	M	ICP-MS (Miller)	Lecture		What is ICP-MS? How does it work?
6	20-Feb	W		Lecture		Applications of ICP-MS, strengths and weaknesses
6	22-Feb	F		Lab	JGB 6.310b	Lab introduction - Demo: solution mode instrument optimization
7	25-Feb	M		Lecture		Sample preparation, data sequence planning
7	27-Feb	W		Lecture		Solution mode analysis - start to finish tour of integrated components
7	1-Mar	F		**Lab 3**	JGB 6.310b	What's in my sample? Semi quant analysis and full spectral scans
8	4-Mar	M		Lecture		Dealing with interferences
8	6-Mar	W		Lecture		Data reduction - assessing quality control
8	8-Mar	F		**Lab 4**	JGB 6.310b	Designing a method; Cal/QC stds/unknown analysis/data processing
			Project Road Check - drafts of TIMS and ICP-MS methods portions due			
		MWF	***March 11-15 Spring Break***			
9	18-Mar	M	Laser Ablation (Miller)	Lecture		What is LA-ICP-MS? How does it work?
9	20-Mar	W		Lecture		Applications of LA-ICP-MS
9	22-Mar	F		Lab	JGB 6.310b	Lab tour - designing an experiment and method
10	25-Mar	M		Lecture		Assessing LA-ICP-MS data quality
10	27-Mar	W		Lecture		Data reduction
10	29-Mar	F		**Lab 5**	JGB 6.310b	Trace element determinations in solids
11	1-Apr	M	MC-ICP-MS (Loewy/ Koleszar)	Lecture		What is MC-ICP-MS? How does it work?
11	3-Apr	W		Lecture		Applications of MC-ICP-MS, strengths and weaknesses
11	5-Apr	F		Lab	JGB 6.310ab	The Isoprobe - Running the machine
12	8-Apr	M		Lecture		Method design (interferences, data acquisition, cup configuration)
12	10-Apr	W		Lecture		Data processing and quality control
12	12-Apr	F		**Lab 6**	JGB 6.310ab	The Isoprobe - Collecting Data
13	15-Apr	M	Gas Source MS (Larson)	Lecture		Principles of gas source light isotope ratio MS and applications
13	17-Apr	W		Lecture		Instrument peripherals and measurement techniques
13	19-Apr	F		**Lab 7**	JGB 5.140	Lab tour - Sample and run log preparation
14	22-Apr	M		Lecture		Optimizing measurement techniques (calibration and linearity)
14	24-Apr	W		Lecture		Data reduction and designing better sampling strategies
14	26-Apr	F		**Lab 8**	JGB 5.140	IRMS data measurement and data reduction
15	29-Apr	M	Noble Gas (Stockli)	Lecture		Noble Gas mass spectrometry
15	1-May	W		Lecture		Noble Gas mass spectrometry
15	3-May	F	**Final Synthesis**			***Last class day (synthesis wrap up of projects and course)***