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

TTh 12:30-2:00, Lecture – EPS 1.126 (or on-line); Lab – JGB 1.132

**Instructor:** Dr. Nathan Miller, Office: JGB 6.104a, Zoom 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 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*  
*Involve me and I’ll understand*  
- Benjamin Franklin

**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:

- 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 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. 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://listserv.syr.edu/scripts/wa.exe?SUBED1=PLASMACHEM-L&A=1](https://listserv.syr.edu/scripts/wa.exe?SUBED1=PLASMACHEM-L&A=1)

**Class Websites:** We will use Canvas throughout the semester: [https://courses.utexas.edu/](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/](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, [https://diversity.utexas.edu/disability/](https://diversity.utexas.edu/disability/)

**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/](https://catalog.utexas.edu/law/academic-policies-and-procedures/honor-system/)

**Safety and Class Participation/Masks:** We will all need to make some adjustments in order to benefit from in-person classroom interactions in a safe and healthy manner. Our best protections against spreading COVID-19 on campus are masks (defined as cloth face coverings) and staying home if you are showing symptoms. Therefore, for the benefit of everyone, this is means that all students are required to follow these important rules.

- **Every student must wear a cloth face-covering properly in class and in all campus buildings at all times.**

- **Students are encouraged to participate in documented daily symptom screening.** This means that each class day in which on-campus activities occur, students must upload certification from the symptom tracking app and confirm that they completed their symptom screening for that day to Canvas. Students should not upload the results of that screening, just the certificate that they completed it. If the symptom tracking app recommends that the student isolate rather than coming to class, then students must not return to class until cleared by a medical professional.

- Information regarding safety protocols with and without symptoms can be found here. If a student is not wearing a cloth face-covering properly in the classroom (or any UT building), that student must leave the classroom (and building). If the student refuses to wear a cloth face covering, class will be dismissed for the remainder of the period, and the student will be subject to disciplinary action as set forth in the university’s Institutional Rules/General Conduct 11-404(a)(3). Students who have a condition that precludes the wearing of a cloth face covering must follow the procedures for obtaining an accommodation working with Services for Students with Disabilities.
Sharing of Course Materials is Prohibited: No materials used in this class, including, but not limited to, lecture hand-outs, videos, 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 promotes cheating. It is a violation of the University’s Student Honor Code and an act of academic dishonesty. I am well aware of the sites used for sharing materials, and 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.

Class Recordings: Class recordings are reserved only for students in this class for educational purposes and are protected under FERPA. The recordings should not be shared outside the class in any form. Violation of this restriction by a student could lead to Student Misconduct proceedings.

COVID Caveats: To help keep everyone at UT and in our community safe, it is critical that students (and faculty and staff) report COVID-19 symptoms and testing, regardless of test results, to the HealthPoint Occupational Health Program (OHP) as soon as possible. Please see this link to understand what needs to be reported. In addition, to help understand what to do if a fellow student in the class (or the instructor or TA) tests positive for COVID, see this University Health Services link.
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 typically due before the next class time, and late postings are not allowed. I will be reading what you write and provide feedback as needed. 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. I encourage you to write these in MS Word (or similar) to take advantage of grammar/spelling tools, then paste into Canvas. Please do not cut and paste long sections from the original articles 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

Grading - 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%); **Tue 20-Oct:** 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 (30-35%): To develop ICP-MS problem solving skills, you are challenged to develop an analytical method project designed to analyze “real” samples. Because of Covid-19 related changes to the semester, we will need to design a single class project rather than individual student projects, but the class project will incorporate both solution and laser ablation analysis for a common sample set. Ideally, we will find a sample set of broad interest for the entire class. The project should focus on developing, testing, and documenting quality control for two methods (solution mode ICP-MS and 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). The project components will be broken up among students, with each responsible for completing and documenting their particular components. You will document the class project in the form of a website, that you will collectively develop as a class over the semester. The project will require analytical time outside of class hours and this must be completed prior to the Thanksgiving break. Work after the break should focus on data analysis and finalizing the website. 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.

**Important deadlines:**
- Tuesday Sep 29 – Rough draft of project proposal due
- Thursday Oct 29 – Final draft of project proposal due
- Thursday Nov 12 – Sample preparation complete; samples must be fully ready for analysis.

**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 10, or T Dec 15).
# Class Schedule of Activities

<table>
<thead>
<tr>
<th>Wk</th>
<th>Day</th>
<th>Date</th>
<th>Topic</th>
<th>Lecture/Lab</th>
<th>Meeting Location</th>
<th>Important Dates</th>
<th>Classes</th>
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</thead>
<tbody>
<tr>
<td>1</td>
<td>Th</td>
<td>27-Aug</td>
<td>Intro &amp; Chem Bkgd; ICP-MS components</td>
<td>1. Course Introduction Pt 1: ICP-MS vs. the world</td>
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<td>2</td>
<td>T</td>
<td>1-Sep</td>
<td></td>
<td>1. Course Introduction Pt 2: Heritage of ICP-MS</td>
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<td>Discussion topic 1 due</td>
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<td></td>
<td>Th</td>
<td>3-Sep</td>
<td></td>
<td>2. Atomic structure, isotopes, ions, ionization; Overview of major ICP-MS instrument components</td>
<td></td>
<td>Discussion topic 2 due</td>
<td>3</td>
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<tr>
<td>3</td>
<td>T</td>
<td>8-Sep</td>
<td>Ionization</td>
<td>3. Sample Introduction: Nebulizer-Spray Chamber-Torch</td>
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<td></td>
<td>Th</td>
<td>10-Sep</td>
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<td>4. Sample Introduction: Plasma Source</td>
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<td>Discussion topic 3 due</td>
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<td>4</td>
<td>T</td>
<td>15-Sep</td>
<td>Ion Sampling</td>
<td>5. Sample Introduction: Vacuum Interface</td>
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<td>Discussion topic 4 due</td>
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<td></td>
<td>Th</td>
<td>17-Sep</td>
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<td>6. Ion Focusing System</td>
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<td>Discussion topic 5 due</td>
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<td>5</td>
<td>T</td>
<td>22-Sep</td>
<td></td>
<td>7. Quadrupole Mass Analyzer</td>
<td></td>
<td>Class Method Project Proposal Draft Due</td>
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<tr>
<td>6</td>
<td>T</td>
<td>29-Sep</td>
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<td>9. Spectral interferences: Isobaric, polyatomic and doubly charged species; Mathemetic correction equations</td>
<td></td>
<td>Class Method Project Proposal Final Draft Due</td>
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<td></td>
<td>Th</td>
<td>1-Oct</td>
<td>Ion Detection</td>
<td>10. Collision/Reaction Cell technology</td>
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<td>Discussion topic 7 due</td>
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<td>7</td>
<td>T</td>
<td>6-Oct</td>
<td>Sample Prep, QA/QC</td>
<td>11. Detectors</td>
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<td>Class Method Project Proposal Final Draft Due</td>
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<td>Th</td>
<td>8-Oct</td>
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<td>12. Sample Prep Considerations &amp; Contamination Control</td>
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<td>Discussion topic 8 due</td>
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<td>8</td>
<td>T</td>
<td>13-Oct</td>
<td>Laser Ablation</td>
<td>13. Analysis of solid materials by LA-ICP-MS</td>
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<td>Discussion topic 9 due</td>
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<td>9</td>
<td>T</td>
<td>20-Oct</td>
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<td>15. Murky questions and review of topics</td>
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<td>Th</td>
<td>22-Oct</td>
<td>Midterm Exam</td>
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<td>10</td>
<td>T</td>
<td>27-Oct</td>
<td>Solution mode: Tuning, unknown ID via FSMS &amp; semiquant analysis, making cal stds, method design for quant analysis, &amp; data reduction</td>
<td>Lab 1. Lab tour; Tuning the Instrument; Solution mode: team unknown identification via full spectral mass scans</td>
<td>JGB 1.132 or Zoom</td>
<td>Class Method Project Proposal Final Draft Due</td>
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<td>Th</td>
<td>29-Oct</td>
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<td>Lab 2. Solution mode: unknown identification via semiquant analysis</td>
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<td>LAB 1 Due</td>
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<td>11</td>
<td>T</td>
<td>3-Nov</td>
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<td>Lab 3. Solution mode: making calibration standards</td>
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<td>LAB 2 Due</td>
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<td></td>
<td>Th</td>
<td>5-Nov</td>
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<td>Team calibration standard making, testing</td>
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<td>12</td>
<td>T</td>
<td>10-Nov</td>
<td>LA-ICP-MS: Tuning, exp design, analysis, data reduction</td>
<td>Lab 4. Solution mode data reduction</td>
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<td>Project Sample Preparation Complete</td>
<td>19</td>
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<td></td>
<td>Th</td>
<td>12-Nov</td>
<td></td>
<td>LA-ICP-MS demo and Iolite</td>
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<td>13</td>
<td>T</td>
<td>17-Nov</td>
<td>Hands-On Class Project Time</td>
<td>Lab 5. LA-ICP-MS data reduction</td>
<td>EPS 1.126</td>
<td>LAB 4 Due</td>
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<td>Th</td>
<td>19-Nov</td>
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<td>Scheduled class project time</td>
<td>JGB 1.132</td>
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<td>14</td>
<td>T</td>
<td>24-Nov</td>
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<td>Scheduled class project time</td>
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<td>LAB 5 Due</td>
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<td></td>
<td>Th</td>
<td>26-Nov</td>
<td>Thanksgiving Break November 25-28 (no class)</td>
<td>Scheduled class project time</td>
<td>Zoom</td>
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<td>23</td>
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<td>15</td>
<td>T</td>
<td>1-Dec</td>
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<td>Scheduled class project time</td>
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<td>Th</td>
<td>3-Dec</td>
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<td>Scheduled class project time</td>
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<td>25</td>
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<td>16</td>
<td>T</td>
<td>8-Dec</td>
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<td>No classes: preparation for student presentation and final paper</td>
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<td>Finals</td>
<td>Th</td>
<td>10-Dec</td>
<td>Class project presentation (will be in this time window)</td>
<td>Class method project website presentation</td>
<td>Zoom</td>
<td>Day TBD within UT Finals Week: Dec 12-14, 16-18</td>
<td>28</td>
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GEO 391 - Class Method Proposal

Due dates: Draft 1 – 9/29/20 (T); Final Proposal 10/29/20(Th); Sample preparation by- 11/12/20 (Th)

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 identify the specific subject in as few words as possible
   - Attract attention to the research hypothesis
   - Clearly reflect the method development objectives
2. Executive summary/abstract - should
   - Describe the proposed research objective or hypothesis (research problem)
   - Describe the method objectives & expected outcomes (problem strategy)
3. Research objective or hypothesis and specific objectives of the ICP-MS/LA-ICP-MS method
   - This section should briefly elaborate the greater research objective that your method will be applied to in order to provide useful data. It should elaborate in more detail what the method will do and specify all of the analytes to be included.
4. Discussion of significance or need (justification)
   - This section should describe why the method is of potential importance and why analytes listed above are specifically important or relevant to the research objective(s)
   - This section should describe what degree of accuracy and precision is required for the method to be useful.
   - This section may present previous or preliminary research bearing on the need for method development.
5. Review of relevant work (literature review)
   - This section should summarize relevant ICP-MS or LA-ICP-MS research applied to similar sample types; recent and state-of-the-art research applicable to your research goal is what you want to include; PlasmaChem Listserv is a possible resource if you have problems finding published research
   - Describe 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
   - This section should outline the working plan for how you will develop and test your method in order to obtain the required data quality. State specifically what you plan to analyze and how you will evaluate accuracy and precision of the data obtained by the method.
   - This section should justify the budget materials and time, however the actual budget should be 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
   - Not likely you can say much in proposal, but good practice to enumerate points of justification and benefits to be derived; draw the reader back to 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. 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 ideally indicates that you are capable of developing the proposed method; emphasize relation of applicable training to expertise needed; avoid points unimportant to this research.
10. References
   - This section indicates the extent to which you’ve explored relevant analytical literature for your subject.

A complete proposal will satisfy the proposal purpose above. Does yours do this?
Home Page

Introduction
- Background
- Research Purpose
- Hypothesis
- Method Goals

Sample Description/Preparation
- Influence on data quality?

Description of Instrument(s)

Experimental Setup (Table)
- Analyte masses
- Internal standard masses/concentration in unknowns
- Secondary (external) standards
- Modes (He, H2, NG) used, and specific reasoning

Instrument Configuration (Table)

Experimental Parameters/Operating Conditions
- Describe how were they obtained?

Optimization
- Instrument Tuning
- Sensitivity on Tuning (e.g., LA tuning – how many cps/ppm were obtained?)
- Oxide production – what oxide levels were measured during tuning?
- How were potential interferences dealt with?

Analytical Sequence
- Demonstrate how quality control was considered

Data Quality
- Drift compensation Accuracy
- Precision
- Signal to noise (analyte intensities for unknowns: background intensities)
- Laser power output variation

Results/Findings
- Did everything work right the first time? Describe the process by which you obtained final results.
- Which analytes/modes worked best?
- How did unknown concentrations correspond to the calibration range?
- Figures are easier to interpret than tables of numbers

Future Work
- Description of how the method worked for your research goal, and what might be explored in future work to better constrain the research problem(s).
- What would you do differently?

Questions
- Entertain questions from your peers about your method project.
GEO 391 Student Project Final Report (5-10 pages)

Should:
1. Follow from your final proposal (e.g., introduction, references) and class presentation
2. **Feature** a well-written methods section
3. Include at least one example (Figure/Table) of how the data obtained addressed the research problem (for examples, see: [http://www.jsg.utexas.edu/icp-ms/projects/](http://www.jsg.utexas.edu/icp-ms/projects/))
4. Include a final results table of concentrations (and/or isotope ratios and ages) limited to analyte modes for which you obtained the highest data quality
5. Include final discussion of how the method worked toward your research goal, and how you would approach future research using ICP-MS tools using knowledge gained from this project.

Methods Section
Don’t reinvent the wheel for the methods section; refer to the literature (you should have found good examples when writing your proposals) to see how methods sections are written. Find examples you feel are high quality and comprehensive; these are more likely to be found in literature specifically documenting a LA/ICP-MS method or technique. Note – there are numerous examples of poorly written methods sections in the literature. **Remember, the broad goal of a methods section is to document what was done in sufficient detail that others could replicate the analytical conditions and can evaluate the quality of the data that was obtained (accuracy, precision).**

**Methods section should include:**
- Analytes that were specifically measured
- Specific instrument(s)/configuration(s) used and where instruments are they located
- Dates analyses were performed
- Description of how the instrument was optimized and sensitivity levels (cps/ppm) obtained across the amu range based on tuning from a standard; describe standard
- Description of how interferences on target analytes were avoided or dealt with?
- Description of how instrumental drift was corrected
- Documentation of detection limits obtained for analytes and typical ranges of measured intensities for analytes (the intensity divided by the detection limit is a measure of the signal-to-noise for analyte measurement)
- Description of the accuracy and precision obtained (based on external reference/quality control standard replicates run over the analytical session).
- Mention/description of the reference standards analyzed for the former?
- Discussion of extent of any matrix effects on the analyses? For solution mode, this is addressed from the extent to which matrix spike recoveries correspond to spiked additions.
- Documentation of specific operating parameters (usually in a table).
- Description of how data were reduced to obtain concentration data (typically ppm or ppb)

**Follow an organizational plan**
For example: Chronological – sample prep, determination of method parameters, instrument optimization, reference to results (tables/figures), documentation of data quality (from statistical analysis of the data obtained during the analytical session(s)).

The ICP-MS lab web site publications tab may be useful for finding some examples of methods sections for our instrumentation. For examples, see: [http://www.jsg.utexas.edu/icp-ms/publications/](http://www.jsg.utexas.edu/icp-ms/publications/)