Syllabus: GEOS F600-F01 Fall 2013 CRN 76346

Introduction to X-ray Spectrometry Ken Severin X5821 • kpseverin@alaska.edu • Room 324 Reichardt

After completing this course the student should be able to critically evaluate electron microprobe and x-ray fluorescence (XRF) analyses of materials. Students will be exposed to the theoretical and practical aspects of obtaining microprobe and XRF analyses. To some extent, having you become familiar with specific instruments and techniques are "minor goals:" In my perfect world I would like you to develop the understanding that no instrument of any kind gives answers to really interesting questions, instead the instrument can only make measurements, and it is critically important to understand the limitations of those measurements. What you do with those measurements is interesting, deciding what measurements to make is hard. I hope that you will appreciate that all measurements come with limitations, and dealing with those limitations is a fundamental part of any investigation.

Lectures will deal mainly with the theoretical aspects of x-ray spectrometry, while labs will focus on the mechanics of obtaining analyses from the electron microprobe and Axios XRF housed in the Advanced Instrumentation Laboratory. At the conclusion of the course the student will present (via a 10 minute talk and 8-10 page paper) an <u>analytical protocol</u> that uses one or both of these techniques. Do not this confused with a project where you are making scientific interpretations based on data you have collected: For GEOS F600 you should be concerned with the quality of the data. To encourage this, you are limited to the analysis of three "real world" samples until you have a draft of your final paper approved. I can't emphasize enough that in the vast majority of cases the quality of the data you need is determined by the problem you are studying, and that understanding the quality of the data is fundamentally important. No one has a big enough checkbook to get "the absolute best data" and rarely is "the absolute best data" definable or needed. You are welcome to use any data you gather in this class for any other classes you are taking (but if you are going to use the data commercially, meaning money is changing hands, talk to me first). Talks are open to the general community, expect written and oral comments from classmates and others. You will be expected to understand both the precision and the accuracy of your analyses, and to understand how your analyses are affected by sample preparation, beam (electron or x-ray)-sample interactions, and analytical conditions.

Lectures: Mondays, 2:15-5:15, 306 Reichardt Building (Geology/Spencer Library unless otherwise noted!) **Labs:** each student will have one scheduled three-hour lab session/week which meets somewhere around the Microprobe Lab, 156A NSF. Labs will be scheduled at the first class. Students will typically need 3-6 hours of additional lab time per week. **The more time you put in with the instruments, the more comfortable you will be with them.**

Grading: Your final grade will be based on:

- 1) Labs (5% Each Total 40%). The maximum possible for each lab will be reduced by 5% a day for each day or portion there of for labs turned in after the due date unless you have received explicit permission for it to be late from me.
- 2) Quizzes (5% each Total 15% Toss the lowest score, but you do have to take them all)
- 3) Oral presentation (10%)
- 4) Written project report (total 20%: 10% final, 5% proposal, 5% outline)
- 5) Class Participation (includes leading discussion on reading) (5%)
- 6) Laboratory Safety, Practice, and Hygiene (10%)

A	96-100	B+	89-93	C+	82-85	D+	73-75
A-	93-96	B	87-89	C	78-82	D	72-73
		B-	85-87	C-	75-78	D- F	70-72 <70

	Tentative Lecture Schedule	Laboratory
Sept. 10	Characteristics/terminology of X-ray spectra,	Lab tour/ Introduction to the microprobe and
	instrumentation XRF overview,	EDS
C + 17	organizational stuff	D I EDC I ' '' ' I '
Sept. 17	X-ray/solid interaction - mass absorption coefficients	Probe - EDS and semiquantitative analysis
Sept. 24	Error Analysis/ Counting Statistics	More Probe - Intro to PFE and WDS
		spectrometers
Oct. 1	X-ray Fluorescence Analysis	WDS-XRF semi-quant
Oct. 8	Electron/solid interaction – implications for	WDS-XRF full-quant
	electron microprobe analysis	
	Quiz 1	
Oct. 15 Ken Gone	IN CLASS ASSIGNMENT - (TBA)	XRF Sample Prep (or TBA)
Oct. 22	Elemental Mapping and X-ray Detectors	PFE- counting statistics
Oct. 29	Instrument Design	Thin Sections / Sample Prep
	Quiz 2	
Nov. 5	More Probe for EPMA	Probe for Windows – Full Quant
Nov. 12	Electron Microprobe Quantitative Methods	BSE Imaging(short optional lab)
Nov. 19	Bence Albe Correction Methods	Projects
	Quiz 3	
Nov. 26	Electron Microprobe Quantitative Methods –	Projects
	what we really do.	
Dec. 3	Paper Review, published and other classes	Projects
	Quiz 4	
Dec. 10	Student Presentations	FINAL PAPER DUE Dec. 17
	Last day to turn in draft of final paper for	
	guaranteed comments.	

Text: Goldstein, JI, Newbury, DE, Joy, DC, Lyman, CE, Echlin, P, Lifshin, E, Sawyer, L, and Michael, JR. 2003. Scanning electron microscopy and X-ray microanalysis. Kluwer Academic / Plenum Publishers, New York. 689 pages plus CD. This is the third edition, and there are some changes from the second. There are several similar books from the same group in the library.

Other resources material – much of the material is duplicated from one text to another, but every author presents things slightly differently. If you have problems with a concept check out the topic in another.

Potts PJ. 1987. Potts PJ. 1987. A Handbook of Silicate Rock Analysis. Blackie. Glasgow, UK. 622 pages. A great reference but WAY pricey (and I think out of print now).

Reed, SJB. 1996. Electron microprobe analysis and Scanning Electron Microscopy in Geology. Cambridge University Press, Cambridge. 201 pages.

Williams, KL. 1987. An introduction to X-ray spectrometry: X-ray fluorescence and electron microprobe analysis. Allen and Unwin, London. 370 pages. Also out of print.

Dr. James Wittke (Northern Arizona University) has a very nice set of notes and references at http://www4.nau.edu/microanalysis/Microprobe/NewOverview.html

Oxford Instruments (http://www.x-raymicroanalysis.com/pages/main/main.htm) has some nice introductory information on both EDS and WDS as well.

LAB and ASSIGNMENT SCHEDULE

	Happens the week of	Due (by 5pm – remember there are penalties for handing things in late!)
Lab 1, Introduction to AIL and the Microprobe	Sept. 9	Lab 1 due Sept 20
Lab 2, Microprobe: Semiquantitative analysis	Sept 16	Lab 2 due Sept 27
Lab 3, Microprobe: Intro to WDS spectrometer	Sept. 23	Lab 3 due Oct. 4
Lab 4, XRF: Introduction	Sept. 30	Lab 4 due Oct. 11
Lab 5, XRF II Quiz 1 in class Oct. 8	Oct 7	Lab 5 due Oct. 25
Lab 6, XRF Sample preparation (I hope!)	Oct. 14	Turn in with lab 7
Lab 7, Microprobe Sample preparation	Oct. 21	Written project proposal due Oct 22 Lab 6-7 due Nov 1.
Lab 8, Microprobe: Counting statistics and homogeneity Quiz 2 in class Oct. 29	Oct. 28	Lab 8 due Nov. 8
Lab 9, Microprobe: Quantitative microprobe in PFE	Nov. 4	Lab 9 due Nov. 15
Quiz 3 in class Nov. 19		
Project Outline	Nov. 19	Written project outline due Nov. 19
Quiz 4 in class Dec. 3		
Project Talks	in class Dec. 10	
Last day for guaranteed review of paper draft	Dec. 10	
Project Paper	Dec. 17	Dec. 17

Safety Quiz (Online at http://www.uaf.edu/safety/training/safety-training-powerpoin/Lab-Safety-Sept-2011.ppt or send me an email saying that you have completed something equivalent or greater.)

Students will conduct themselves in accordance with the "Student Code of Conduct" as put forward in the applicable University of Alaska Fairbanks Catalog.

Disabilities Services: The Office of Disability Services implements the Americans with Disabilities Act (ADA), and insures that UAF students have equal access to the campus and course materials. The assistants and I will work with the Office of Disabilities Services (203 WHIT, 474-7043) to provide reasonable accommodation to students with disabilities.

This syllabus is a contract between you as a student and me as the instructor and it cannot be changed after the first lecture. If you do not wish to be follow it then do not take the class.