



In situ monazite and xenotime geochronology by electron microprobe analysis (EPMA) at the University of Colorado, Boulder

January 16th, 2017

Lab description

The CU Boulder Electron Microprobe Laboratory hosts a JEOL JXA-8230 electron probe microanalyzer (EPMA), a carbon and metal evaporator for coating, and a thin section preparation shop. Our new EPMA was installed in March 2016, and is tailored for major and trace element analyses of solid materials (mineral, glass, alloy, etc.) at the micron-scale. Among numerous applications, the CU Boulder EPMA laboratory offers the ability to non-destructively date *in situ* REE-phosphate (monazite or xenotime) using the U-Th-Pb_{total} dating technique (Montel et al., 1996; Chem. Geol. 131, 37-53). The EPMA can provide high spatial resolution down to 1 μm , permitting to date precisely and accurately micron-scale domains or overgrowths within single grains. Samples as young as 500 Ma can be dated easily with this technique, with 5 to 20 myr resolution depending on the Pb-content. Younger samples (100-500 Ma) are subject to larger errors, but can be dated if they are rich-enough in actinides (Th, U).

REE-phosphates often record more information than zircon or other datable minerals, and are found in a large variety of rock types. Monazite dating by electron microprobe can be applied to wide range of problems, for instance:

- Metamorphic monazite and xenotime are common in metapelites, many quartzo-feldspathic gneissic rock types, and rarely mafic and ultramafic rocks, and can help constrain the timing of tectono-metamorphic processes. Monazite grains often contain multiple overgrowth (or alteration) stages, corresponding to a variety of potential processes: e.g., diagenesis, prograde or retrograde metamorphic reactions, deformation event(s), etc.
- In magmatic systems, silicic rocks (granite, rhyolite) are most likely to contain monazite, and can be helpful in determining the crystallization age of the unit, as U-Th-Pb dating of monazite has a high closure temperature beyond 900°C even for grains as small as 10 μm (Cherniak, 2004; Geochim. Cosmochim. Acta 68, 829-840).
- In sedimentary rocks, monazite can be used, similarly to zircon, as a provenance analysis tool. Under some circumstances, low-grade monazite (200-400°C) can grow in the sediment, and can potentially be used as a tracer for diagenesis, alteration by hydrothermal fluids, or low-grade metamorphism (see for instance Allaz et al., 2013; Am. Min. 98, 1106-1119).
- Under certain circumstances, fluids can potentially dissolve and reprecipitate monazite. Therefore, it has potential for application to a range of other fluid-related geological problems (e.g., mineralization or remobilization of a mineral deposit, metasomatism).

Time frame

If the student already knows how to use a scanning electron microscope (SEM, TEM) or an EMPA, he/she will participate in a 1-day basic training session on the use of our specific instrument, which will include the theory behind monazite U-Th-Pb dating. A student with no or limited experience on such instruments will be encouraged to take the 3-day workshop option. This workshop, possibly shared with 3 or 4 other students, will include basic theoretical principles behind SEM and an EMP usage, a significant amount of practice on the

instrument, and a special focus on monazite geochronology by EPMA. The student will have the opportunity during the 1- or 3-day workshop to think about his/her own project, and how to best approach the project.

The complete analysis of one sample can usually be done in two to three days depending on the complexity (e.g., zoning and/or generations present) of the analyzed REE-phosphates. Per sample, the following will be done:

- (1/2 day) Element mapping to search for REE-phosphate grains in a thin section. A typical project would have at least one sample mapped by this or a similar fashion to insure feasibility of the project prior to the student's arrival.
- (1/2 day) Detailed element mapping of individual REE-phosphate grains (10-20 per sample) to determine the intra-grain compositional variation. Time for mapping one individual grain can vary from 10 minutes for 20-50 μm grains to 30-60 minutes for grains over 200-500 μm .
- (1 to 2 days) Quantitative analysis: ~20 to 30 minutes per analysis point and 4-8 points per domain. A domain is defined as a compositionally homogenous area of a monazite grain. The complexity of a sample (i.e., the number of different domains found in one sample) is impossible to predict. Most of the time, 1 day is sufficient. Note that the data obtained for each analyzed domain is a complete chemical analysis. Thus, along with the spatial and microtextural context from the mapping phases of the project, much more information is acquired besides a "date." These data can be quite valuable for ultimately relating dates to petrogenetic processes.

Calibration and standardization of the EPMA (1/2 day), is done with the student the first day of the analysis session. Full thin section mapping is done overnight (2 to 3 samples mapped per night). Depending on the complexity of the sample, notably its geological history, a couple more analysis days might be required for additional grain maps and/or quantitative analysis points. We anticipate that the student will bring no more than 4 or 5 samples for his/her project (assuming 1 week of analysis time), although exceptions can be made if the samples are expected to have a simple geological history or if the student desires more than 1 week of analysis time.

Many analytical tasks will be conducted in automated mode, leaving thus ample time for discussion with the student on the treatment of the results, and discussion of the data interpretation. Once the data are acquired, the student will return home with the acquired data and a copy of the software to permit further data reprocessing at his/her home institution. If desirable, the student can stay a few more days on site to further discuss the results and their treatment. The laboratory manager will remain available any time after that to discuss and help interpret the results. In addition, while the student is on site at CU, they will be welcome to participate in regular group meetings with CU students working with Drs. Mahan and Allaz, and to share their research experiences with the group.

Analytical costs

Full EPMA microprobe training (3-day workshop) *	\$1,000	
Quick training on the electron microprobe (1-day) **	\$400	
Sample preparation (thin section, if required)	\$20	per sample
EPMA calibration and standardization (6 hours)	\$500	one-time charge
Full thin section mapping (5 hours)	\$150	per sample
Monazite or xenotime mapping (10-20 grains)	\$150 to \$300	per sample
Quantitative analyses (24 hours)	\$800 to \$1,000	per sample
Estimate total (per sample)	\$1,200 to \$1,500	

* Include 2 days of basic usage of electron microprobe (theory & practice) and one day of training specific to monazite dating. A certificate of attendance will be provided at the end of this training.

** Only applicable to student who already have knowledge of scanning electron microprobe. This training will focus on monazite U-Th-Pb dating by electron microprobe.

Preparation for Visit

The student must come with at least one thin section per sample, and it is encouraged that at least one sample must have been evaluated for the presence of the target REE-phosphates prior to the visit. Such a pre-visit evaluation can be done by our lab if the student desires - contact us for more information. The thin sections must have a very-good final polishing, as data quality will heavily depend on it. Each sample should have been carefully investigated under the petrographic microscope prior to the student's visit. The student should contact us, if the preparation of well-polished thin sections is required.

Although not required, we encourage the student to learn more about electron microscopy prior to the visit. Several resources are available freely on the web or at your local library. Some references are listed on our electron microprobe website at:

<http://geode.colorado.edu/~jallaz/index.php?page=microprobe>

Laboratory staff

The electron microprobe laboratory at CU is run by Associate Researcher Julien M. Allaz, and supervised by Prof. Kevin H. Mahan. At least one graduate student laboratory assistant is also available for help on the use of the electron microprobe or the interpretation of age data. Julien will be the main point of contact for the student, and will continue providing support to the student on data processing and interpretation after his/her visit.

Data processing & interpretation

At the end of the visit, the student will also have learned about data processing, presentation, and interpretation. A copy of the software will be given to the student to permit reprocessing of the data at his/her home institution. While in the lab, the student will be able to process data "on the go", within minutes after the acquisition. Julien will be available to help the student in the data processing. Following data acquisition, the student will have the opportunity to discuss with laboratory staff how to present the results in a graphical way (e.g., age plots, element plots, chondrite normalization diagrams, etc.), and how to interpret the results. Since the analyses are done *in situ*, emphasis will be put on the potential relationships between the analyzed dates, microstructure, and the relation of monazite/xenotime with other rock forming minerals, in order to provide a robust geochronological interpretation.

Expected lab availability

The current wait time for our laboratory is 1 to 2 months for monazite dating projects. An additional delay of a month might apply, if thin sections need to be prepared in house.

Contact information

If you are interested in using the electron microprobe at CU Boulder, or if you have any questions regarding a potential collaboration, please, contact either:

Julien M. Allaz: julien.allaz@colorado.edu

Kevin H. Mahan: kevin.mahan@colorado.edu