AGeS3 – Advancing Geochronology Science, Spaces, and Systems
University of Kansas – Isotope Geochemistry Laboratories (IGL):
ID-TIMS and ID-ICPMS Facilities for Geochronology and Thermochronology
17 January 2023

Lab Description

The Isotope Geochemistry Laboratories (IGL) at the University of Kansas consists of

- a sample preparation laboratory for mineral separation,
- a class 10/100 clean lab for low-blank radiogenic Pb, U, Th, Sr, Nd, and other trace element work, and
- a mass spectrometry lab with
    - two thermal ionization mass spectrometers (TIMSs) and
    - an Element2 sector-field inductively coupled mass spectrometer (ICPMS) with both laser ablation and solution-based sample introduction systems

Sample Preparation and Mineral Separation

The sample preparation laboratory contains standard rock crushing equipment, including a hydraulic rock splitter, a Braun Chipmunk jaw crusher, a Bico disc mill, a ball mill, and a Gemini (similar to Wilfley) water table for hydrodynamic separation of coarse crystalline and volcanic rocks. The crushing facilities are in a dedicated room equipped with fast, large-volume air extraction for controlling dust, and are located beside a separate room with a wide variety of rock saws. More rock sample preparation facilities are available in the Rock and Thin Section Preparation lab, staffed by a full-time technician and offering thin sections, epoxy mounts, and carbonate/feldspar stains.

Additional mineral separation facilities are housed in the new IGL labs in Slawson Hall at KU. These include a Frantz Model L-1 magnetic separator and an exhausted fume hood dedicated to safe heavy liquid (bromoform and methylene iodide) separations. For extracting small, heavy minerals from clay-rich samples, the laboratory also has a set of blenders, large beakers for soaking with acid baths or an iodine solution, and an ultrasonic clay separator (Hoke et al., 2014). For smaller samples, there are a several sizes of ceramic and steel mortar and pestles. The lab also has a variety of large working distance microscopes, including a Leica S9 D stereomicroscope with an integrated digital camera system, and petrographic microscopes.

Clean Laboratory

IGL operates a large two-room class 10/100 clean lab facility for trace metal and isotope geochemistry. Routine work includes low-blank U-Pb, U-Th, Sm-Nd, Sr, and common Pb analyses. The facility operates under continuous HEPA-filtered positive pressure and contains two eight-foot, two six-foot, and one five-foot exhausted laminar flow benches. A Milli-Q IQ 7000 Ultrapure Water System provides 18.2 MΩ-cm water through a Q-POD dispenser, and all acids are distilled in-house by two PFA Savillex DST sub-boiling stills, producing reagents with less than 1 pg/g total Pb, U, and Th blank. Ion exchange column chemistry utilizes custom lab-made equipment. KU IGL employs a variety of in-house isotopic tracers as well as the EARTHTIME U-Pb tracer.

Mass Spectrometry

We have two TIMS instruments in our laboratory. One is a new Isotopx Phoenix with extended geometry, nine Faraday collectors equipped with 10^{11} and 10^{12} ohm resistors, and one Daly ion counting system. The Phoenix is used primarily for high-precision U and Pb (zircon), Sr, and Nd isotopic analyses. Dynamic Sr and Nd analyses on the Phoenix have internal and external precision of better than ± 5 ppm (1RSE) evaluated using NBS987 and JNd-i reference materials. The Daly ion counting system on the Phoenix is linear to > 1 M cps for Pb, U, and uranium oxide as measured by NBS981 and U500 reference materials. The second TIMS is an 8-collector, 20-sample turret VG Sector 54. This older instrument can produce accurate Sr and Nd analyses with typical internal precision of ± 25 ppm. All instruments have UPS and onsite generator power backup. The TIMS instruments are supported by a DG60 degas bench capable of degassing 60 TIMS filaments as well as a dedicated benchtop laminar flow hood area for loading.

The ICPMS laboratory consists of a Thermo ELEMENT2 high resolution sector-field ICP-MS, coupled with a state-of-the-art small footprint ATL ArF Excimer laser with 193 nm wavelength and an ultra-short ~4 ns pulse length to ensure minimal sample heating and independence of ablation characteristics from mineral color. For solution ICP-MS analyses, the ELEMENT2 utilizes a fully automated, integrated ESI autosampler and glass and Teflon cyclonic spray chamber as well as an HF-capable Apex Omega desolvating nebulizer for dry plasma. Sensitivity for U and Th in low resolution mode at a sample aspiration rate of 125 µL per minute in solution mode is ~1.2 Mcps/ppb using the cyclonic spray chamber and a platinum guard electrode, and ~15 Mcps/ppb with < 0.5% UO_2 production using the Apex Omega DSN. For more information about the ICPMS facility's laser ablation capabilities, please refer to its separate AGeS facility description. The LA-ICPMS, under the direction of Andreas Möller, is often used as a pre-screening and reconnaissance analytical tool before clean lab and ID-TIMS work.

Expected Time Frame

Typical projects take 2-6 weeks for 1-5 samples and interested students should inquire 2-6 months ahead of a potential visit, though shorter timescales can sometimes be accommodated for routine (e.g., zircon U-Pb) work. The expected time frame depends on the scope of the project, whether mineral separation is required and if so, on the samples (typical: 1-2 days/sample); whether imaging is required after mineral separation (e.g., CL imaging for polished zircon grain mounts), the level of complexity of subsampling after imaging, clean lab protocols employed (typical: 3-15 days per project, add 3 days for annealing zircons if using CA-TIMS), and whether the project will employ the TIMS, ICPMS, or both (typical: 3-5 hours per sample on TIMS, 10-15 samples/day by ICPMS). Projects including method development may take additional time. Students are encouraged to discuss expected time frames with lab personnel.

Preparation for Visit

Prospective student visitors should contact IGL lab personnel as early as possible, ideally in the planning stages before sampling and fieldwork, to design an experiment that both addresses

and is capable of answering your research questions. If large samples need to be shipped ahead of student arrival, please arrange shipment with IGL lab personnel.

Analytical Costs

Sample preparation costs depend on the type of sample and the extent of sample handling required, but usually run from $200 to $600 per sample. Analytical costs vary by instrument, isotope system, and also by number of analyses needed. For ID-TIMS samples, 5-10 U-Pb analyses (e.g., accessory minerals) are usually required at a total cost of $1500-2000 per sample, including the cost of mineral separation, clean lab work, and mass spectrometry.

For solution ICPMS trace element as well as isotope dilution U-Th ICPMS samples, the cost is $800 per day of ICPMS time. A day of ICPMS time is usually enough for 25-30 unknown analyses, or 12-15 paired U and Th samples. For laser ablation ICPMS analyses, often useful for pre-screening before TIMS, the charge is $1425/day with an average of ~250-330 spots/day.

Laboratory Staff

KU's Isotope Geochemistry Laboratory, including the ID-TIMS and ID-ICPMS facilities, is directed by Doug Walker, Andreas Möller, and Noah McLean and managed by Joe Andrew. Mineral separation is also performed and overseen by Luis Aparicio. Its greatest assets are its rotating cast of graduate and undergraduate researchers.

Data Processing and Interpretation

As part of an AGeS3 project, students are expected to get training in and participate in all analytical steps, including data processing and interpretation. For U-Pb ID-TIMS, this includes training on TIMS software, the open-source Tripoli and ET_Redux programs for data reduction, visualization, and uncertainty propagation, and data archiving at the geochron.org database. Many KU IGL projects involve a component of technique development and are accompanied by new code written in MATLAB or a student's chosen language, in which case the student also receives training in modern scientific programming techniques including testing and version control.

Diversity, Equity, and Inclusion

KU IGL is committed to providing a supportive lab atmosphere that is engaged, respectful, and inclusive. Geology and geochronology suffer from an ongoing lack of diverse representation in race, gender, disability, age, and socioeconomic status. We actively recruit underrepresented minorities in all capacities and foster inclusion by respectfully addressing concerns, providing clear and reasonable expectations, and facilitating trust, collaboration, community, and belonging.

Contact Information

If you are interested in potential collaboration, please contact:
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