

AIRIE Program, Colorado State University

A facility for Re-Os (rhenium-osmium) isotope geochemistry and Hg (mercury) concentration data

OVERVIEW – Description of AIRIE Laboratory Capabilities and Analytical Facility

Welcome to the AIRIE Program (www.airieprogram.org)! The AIRIE Program has pioneered the Re-Os dating of molybdenite and other sulfides, for example, arsenopyrite and pyrite. **Re-Os dating under the AIRIE Program has a strong focus on resource geology: metals (sulfides) and hydrocarbons (bitumen and oil).** AIRIE also routinely analyzes black shales and other organic-rich sedimentary rocks to provide ages for the Geologic Time Scale, including mass extinctions and other anomalies in the sedimentary record.

Established in 2019, **AIRIE-Hg (mercury)** provides an added expertise, with the powerful combination, Hg-Re-Os, to query present day and paleo-environments. Thus, we combine the time component (Re-Os) with Hg anomalies in the geologic record. AIRIE-Hg provides Hg concentrations for just about any media, from rocks to minerals, to fish bones, to quantifying Hg contaminants lurking in soils, marine and lake muds, and plant media.

The AIRIE laboratory facility features two labs for wet chemistry, one for high Re samples and the other for low Re samples. AIRIE has two TIMS (Thermal Ionization Mass Spectrometry) Triton machines for measurement of Re and Os isotopic ratios. Mercury (Hg) analyses are carried out on a state-of-the-art DMA-80 evo Direct Mercury Analyzer (Milestone).

Expression of Interest

Contact AIRIE Program Director, Holly Stein (holly.stein@colostate.edu)

Relevant Laboratory Personnel

Dr. Prof. Holly Stein, Senior Research Scientist and Professor, Founding Director, AIRIE Program

Mr. Aaron Zimmerman, Research Associate III, Laboratory Manager, AIRIE Program

Dr. Gang Yang, Research Associate IV, AIRIE Program

Collaboration

Essentially all projects carried out by AIRIE, whether NSF or industry funded, highlight the spirit of collaboration. That is, we are not only producers of data, but intellectually engaged partners in the interpretation of those data, thereby bringing data to full use by collaborating partners. Our engagement starts from the sampling stage to assure selection of the most promising materials for the project. For over two decades, we have worked with students and academic groups around the world. From the outset, our goal has been to educate others about the value and power in isotope geology. Since our inception, the AIRIE mission statement has withstood the test of time. To an already encompassing mission statement, AIRIE members are actively engaged in DEI activities and reaching out.

Preparations for Contact and Visit

The applicant should have a fundamental grasp on Re-Os isotope geochemistry based on reading of the literature. AIRIE Program director, Holly Stein, will direct the student to relevant reading as preparation for the student's visit to AIRIE. All samples for Re-Os analytical work must be sent *in situ* in their host rock. Description of the geologic context of the mineral to be dated is a critical part of the Re-Os dating process. All mineral separates will be acquired on site at AIRIE.

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Analytical Costs

The AIRIE Program is supported entirely on soft-money, grants and contracts. Colorado State University provides no salary or operational support for the AIRIE Program, so costs for a project must be covered by external users, and any funds the PI partner and/or the AIRIE Program may have available to leverage at the time of the project. Feasibility and cost for a proposed project should be discussed ahead of proposal submission.

The student PI of a funded project is encouraged to spend some time at the AIRIE labs to gain firsthand experience in a Re-Os isotope facility. A student collaborating with AIRIE arrives to a fully functioning laboratory with calibrated Re and Os spikes, including an array of hybrid ^{185}Re -double Os (^{188}Os - ^{190}Os) spikes to address molybdenites, and single ^{185}Re and ^{190}Os spikes to address sulfides, shales, and hydrocarbons. The AIRIE laboratories are maintained in peak functioning order. Blanks are run routinely with every batch of samples and monitored over the longer term. The protocols for dating sulfides and shales, and bitumens and oils, are in place and routinely employed. Colorado State University requires that a cost recovery plan be in place and updated yearly. Our cost for providing a molybdenite age is \$2400, with an additional \$500 per sample for the cost of AIRIE staff to train and shepherd a guest student in the labs. Costs for other sulfides and shale-based projects, including their feasibility and samples needed, should be discussed in advance. Covered under the analytical costs are mineral separations, a detailed report with photos, information on methodology, and a publication ready data table.

Scheduling

For a project involving molybdenite dating, the success rate is essentially 100%, and for 5 samples, a time frame of 6-8 weeks is suggested which covers the time for any second analyses needed to optimize spiking. Scheduling is flexible and presents no issues. For other sulfides or bitumen suites, following feasibility and approval by all parties, the timeframe to complete all analytical work may be 2-3 months.

Time Frame

The time frame needed for a project is highly variable, depending on whether an age is derived from a single sample (e.g., molybdenite), or using the isochron approach (multiple separates analyzed, each providing a point to form an isochron). Interested users should contact the AIRIE Program director (holly.stein@colostate.edu) in advance to discuss the proposal so that the feasibility of a project, the funding, needed timeframe, and the necessary samples are aligned with project goals.

Example of Procedure – Re-Os Dating of Molybdenite

A molybdenite is targeted in a rock sample, and extracted using a diamond-tipped, slow speed drill bit after careful documentation of the molybdenite occurrence and paragenetic relationships. Carefully weighed powder, isotopically distinct ^{185}Re - ^{188}Os - ^{190}Os "double spike", and inverse *aqua regia* are introduced in a borosilicate ampoule (Carius tube), sealed, and heated at 250 °C for 12 hours. Oxidation and equilibration of molybdenite and spike Re and Os upon molybdenite dissolution ensures accurate concentration data independent of analytical yield. Osmium is separated from the solution by solvent extraction into chloroform followed by back-extraction into hydrobromic acid (HBr). Subsequent micro-distillation provides a high purity Os split ready for mass spectrometry. Rhenium is extracted from the remaining inverse *aqua regia* solution by anion exchange chromatography and ready for mass spectrometry without further processing. After loading onto outgassed Pt filaments, Re and Os are measured on Triton thermal ionization mass spectrometers. Barium activators enhance ion yield

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resulting in Re intensities allowing simultaneous Faraday cup detection and Os intensities requiring either peak-hopping secondary electron multiplier or simultaneous Faraday cup collection depending on signal strength. Both Re and Os are analyzed as oxides with ratios corrected for oxygen isotopic systematics. The isotope dilution equation utilizing sample weight, spike weight, measured ratios, background "blank" information, and spike calibration data provides rhenium, radiogenic ^{187}Os , and common Os concentrations used to determine the molybdenite age with errors from each variable propagated and fully reflected in the reported uncertainty on the Re-Os age.