

This is a brief newsletter distributed by email to update SEQUOIA users and enthusiasts regarding the status of the instrument. We would like to share with the SEQUOIA user community the ongoing upgrades occurring at the instrument, science highlights, and the peer-reviewed publications and theses resulting from measurements at SEQUOIA.

Please feel free to share feedback with the User Office or your local contact regarding your experience as a user at the Spallation Neutron Source (SNS) and High Flux Isotope Reactor (HFIR) facilities. This can be done by completing the survey you receive electronically from the User Office after experiments or contacting your local contact or the User Office directly. Please do not hesitate to contact the beamline instrument scientists for any assistance in preparing beam-time proposals or if you are having difficulties with data reduction or analysis.

Staff Updates

The SEQUOIA team has bid farewell to Lacy Jones as a cross-trained SEQUOIA scientific associate (SA). She helped at SEQUOIA as required and bravely led the efforts of the now-completed SEQUOIA vacuum



upgrade project. Lacy joined the SNS team in March 2013, when she worked as an SA for the commissioning of the VISION instrument. After VISION entered the user program, Lacy transitioned to working as an SA at ARCS and SEQUOIA in January 2015. She has recently completed a second master's degree in industrial and systems engineering at the University of Tennessee. She has transitioned to a position in the Neutron Technologies Division as the project management specialist for HFIR and SNS instrument upgrades within the Project Management Group.

Experiments And Publications

The last SEQUOIA newsletter was delivered in January 2017. Since then, run cycles 2017-A, 2017-B, and 2018-B have been completed. We ran 43 experiments in 2017 and 37 experiments in 2018. The SNS had the inner reflector plug (IRP) replaced in the first half of 2018. The poisoned decoupled water moderator for SEQUOIA was also replaced during the IRP outage. The new moderator has improved SEQUOIA's lineshape on the negative energy transfer side of the spectrum. The accelerator has been operating very well with power outputs as high as 1.4 MW.



Intensity as a function of energy transfer for a 5 × 5 cm vanadium plate measured at SEQUOIA using an incident energy of 55 meV in a high-resolution chopper configuration. The two curves compare the moderator performance before (July 2017) and after (May 2018) the changing of the IRP at SNS and its corresponding moderators. There is a small additional peak intensity due to the new moderator, but there is great improvement in energy resolution at negative energy transfers.

Both 2017 and 2018 saw increases in the number of publications of SEQUOIA measurements. SEQUOIA had 20 peer-reviewed publications in 2017 and 28 peer reviewed publications in 2018. The total of 48 peer-reviewed publications produced in 2017 and 2018 is greater than the number produced in the prior 3 years combined (2014–16). Congratulations to all the authors.

We also have had five PhD dissertations that included SEQUOIA measurements. We know many students have been to the beamline and are working hard to finish their research projects.

A large number of the presentations given at the meetings of the American Physical Society in March 2017 and 2018 and at the 2018 American Conference on Neutron Scattering featured SEQUOIA results. We know of other publications that are currently in review, and we expect 2019 to be another productive year for the instrument. A list of SEQUOIA publications is available at <u>http://neutrons.ornl.gov/sequoia/publications</u> and within the PuSH database, which is available at <u>http://neutrons.ornl.gov/publications</u>. We have also listed the 2017–18 publications at the end of the newsletter. Publications that include SEQUOIA results need to be accounted for by Oak Ridge National Laboratory (ORNL) and our sponsors. Please let us know if your SEQUOIA publication or thesis is not listed.





Upgrades

The SEQUOIA vacuum upgrade project was completed in the spring of 2018. The SEQUOIA detectors reside within a vacuum vessel that is 6 m tall and that remains at a vacuum of less than 1x10-6 Torr (equivalent to a low-earth orbit) for months of continuous operation. There are no windows between the sample position and the detectors, thus providing a very lowbackground environment for measurements.

The vacuum upgrade project included the installation of one turbopump on the sample chamber and two turbopumps on the detector chamber for greater pumping capacity, pumping redundancy, and more accurate leak checking of the vacuum system. The project eliminated safety concerns with the location of low-voltage control panels and high-voltage power panels and it removed a nonstandard roughing pump from a radiation materials area. The upgrade project also provided SEQUOIA with standardized gauging and a standardized interface to improve the ability of the SNS vacuum group to troubleshoot or debug the system if required. Pumpdown times of the detector tank have been reduced from 15 hours to 9 hours, and pump-down times of as low as 7 minutes have been recorded for the sample tank (for sample changes).

A scattered-beam radial collimator has been approved by the Neutron Sciences Directorate at ORNL to proceed as soon as funding is available for its purchase. The radial collimator will reduce the background from complicated sample environments. Detailed Monte Carlo studies of various collimator configurations have been made and indicate an improvement in the signalto-noise ratio for measurements that make use of cryomagnets, liquid helium cryostats, and radiative furnaces. The radial collimator will be permanently installed in the scattered beam within the detector tank.

SEQUOIA has also purchased four additional eight-pack linear-position-sensitive He-3 detector modules. SEQUOIA has only 60% of its available detector complement installed. There is currently room to install 76 additional eight-pack detector modules. The installation of the four additional modules will provide a small increase in detector coverage for wave-vector transfers out of the scattering plane. The additional detectors will also serve as live spares if a currently installed detector develops issues.

If you have any suggestions for instrument upgrades that would improve the measurements you perform for your research program, please do not hesitate to share them with the beamline staff.

Science Cross Section

We highlight here two publications that span the missions of the SEQUOIA instrument: measuring hydrogen and magnetic excitations in condensed matter systems.

Two-dimensional magnetic

excitations in Sr₂IrO₄

-by Matthew Stone

Measurements of the guasi-two-dimensional spin-orbital Mott insulator Sr₂IrO₄ were made at SEQUOIA. Despite weak to intermediatestrength spin-orbit coupling, Sr₂IrO₄ has been well described by an effective S=1/2 Heisenberg Hamiltonian with a magnetic bandwidth of approximately 200 meV. Neutron scattering measurements of iridates are challenging due to the large absorption cross section of iridium nuclei and their typically small ordered moment, approximately 0.25 μ_{B} for this case. The issues were overcome on SEQUOIA by using a thinplate composite of coaligned crystals (1.1 g) that exploited the flat-plate geometry of the as-grown crystals. Absorption effects were minimized by measuring volumes of reciprocal space using a limited range of sample rotation which kept the incident wave-vector mostly normal to the plate sample. The data confirm the two-dimensional nature of the magnetic Hamiltonian; complementary CNCS (Beamline 5 at SNS) measurements with $E_i = 3.32$ meV provided improved energy resolution that allowed a gap of less than 1 meV in the excitation spectrum to be quantified. The results were published in S. Calder et al., Physical Review B Rapid Communications 98, 220402(R) (2018).



Background subtracted inelastic neutron scattering of Sr_2IrO_4 measured using SEQUOIA's high-flux chopper. The upper right panel shows the intensity as a function of the (H00) and (00L) reciprocal lattice directions integrated about K = 0+/-0.04 rlu (reciprocal lattice units) and for energies between 4 and 12 meV (*E*i = 20.5 meV). There is no dispersion as a function of *L*. This demonstrates the two-dimensional nature of the excitations and allows one to integrate over the *L*-axis from 0 to 7 rlu to examine the magnetic scattering in greater detail. The left main panel shows the steep magnetic mode coming out of the (-1 0 0) wave-vector for data integrated with K = 0+/-0.04 rlu (*E*i = 60 meV). The right main panel shows the same mode coming out of the (0 1 0) wave-vector integrated for H = 0+/-0.04 rlu (*E*i = 60 meV). Additional results are published in S. Calder et al., Physical Review B Rapid Communications 98, 220402(R) (2018). Data were collected at T = 10 K and an empty can background subtraction was used for all of the data presented.

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Complexity of intercalation in an active layered material: Destabilization of urea by twodimensional titanium carbide

-by Alexander (Sasha) I. Kolesnikov

MXenes are a new class of two-dimensional materials with properties that make them important for applications that include batteries, capacitive energy storage, and electrocatalysis. Intercalation in these compounds is critical for many applications. Therefore, understanding the interaction between MXenes and intercalants is crucial for using MXenes as pseudocapacitor electrode materials.

We investigated urea interaction within

a titanium carbide-based MXene using inelastic neutron scattering (INS) to probe the state of intercalated species. Infrared (IR) spectra of the urea-MXene could not be obtained because the MXene materials are black and strongly absorbing. To get the best energy resolution, we measured the INS spectra with four incident energies, Ei = 30, 110, 250 and 600 meV, selected by the high-resolution Fermi 2 (Ei<200 meV) and high-flux Fermi 1 (Ei>200 meV) choppers at SEQUOIA. SEQUOIA's energy resolution and dense forward detector coverage are valuable in the study of materials containing hydrogen (which has large mean-squared displacement, u_{H^2}) to get information on high-energy modes; only direct geometry spectrometers can provide access to low

momentum transfer, Q, at high energies, thus resulting in reasonable Debye-Waller factors, $exp(-Q^2u_{H}^2)$.

By comparison with reference materials, we found that under intercalation conditions urea decomposes readily in contact with Ti_3C_2 , leading to intercalation of ammonium, which was observed by INS, and evolving carbon dioxide, which was detected by IR spectroscopy. Reactive molecular dynamics calculations provided atomistic insights for reaction pathways. These results show that the active metal sites and strongly acidic/basic moieties in MXenes could serve as catalytic centers that destabilize the intercalating species.



INS spectra for urea-treated MXene and an NH4+intercalated MXene reference sample show excellent agreement for the NH4⁺ modes. ReaxFF was used to provide insights for pathways for urea decomposition on MXenes. INS and IR measurements showed that urea interaction with MXene results in ammonium intercalation and CO₂ evolution. Data are from S. H. Overbury et al., Journal of the American Chemical Society 140, 10305 (2018).

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Future Experiments

Thanks for making 2017 and 2018 great years at SEQUOIA. The next proposal call for run cycle 2019-B beam-time is anticipated to to close at 11:50 a.m. on March 27, 2019 (<u>https://neutrons.ornl.gov/users</u>). We have examined trends in prior requests for beam-time at the instrument. Based on the last six run cycles of SNS, the average number of experiment days for a single-crystal experiment at SEQUOIA is 4.5 days. The average number of days for a powder experiment at SEQUOIA is 2.6 days. Please do not hesitate to discuss your proposals with beamline staff prior to submission.

2017 and 2018 SEQUOIA Publications

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2017 and 2018 SEQUOIA theses

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2 K. Ramic, "From experiments to DFT simulations: Comprehensive overview of thermal scattering for neutron moderator materials." PhD Dissertation, Rensselaer Polytechnic Institute (2018).

3 M. Zhu, "Neutron scattering studies on correlated transition-metal oxides." PhD Dissertation, Michigan State University (2018).

4 C. W. Chapman, "Thermal neutron scattering evaluation framework." PhD Dissertation, Georgia Institute of Technology (2017).

5] Z. L. Dun, "From Pyrochlore to the Tripod Kagome Lattice: Magnetism of new compound family A₂RE₃Sb₃O₁₄ (A= Mg, Zn; RE = Pr, Nd, Gd, Tb, Dy, Ho, Er, Yb)." PhD Dissertation, University of Tennessee (2017).

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